

1982 - 1984

PERFORMANCE REPORT

FROM

WATER QUALITY SECTION

VOLUME 2

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Ministry
of the
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MOE

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1982 - 1984
PERFORMANCE REPORT
FROM
WATER QUALITY SECTION

VOLUME II

Water Quality Section
Laboratory Services Branch
Ministry of the Environment

*** NITROGEN-AMMONIA PLUS AMMONIUM ***

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 01/06/76
Supervisor : F. Tomassini Units : ug/L as N
Sample Type/Matrix: Streams, Lakes, Precipitation

SAMPLING:

Quantity Required: 50 mL
Container : Plastic (polystyrene)

SAMPLE PREPARATION:

Samples are filtered through 0.45u membrane filters.

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the filtrate via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance : 0.40 at the 1000ug/L as N level.

N.B. Nitrate plus nitrite is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W) : 1

Detection Criterion (T): 5

CALIBRATION:

BL plus 4 standards

CONTROLS:

Calibration : LTBL plus 4 standards, eg, QCA

Drift : BL plus 1 standard

NITROGEN - AMMONIA PLUS AMMONIUM

QUALITY CONTROL DATA FROM 13/01/82 TO 01/06/82

LAB: Dorset

Analytical Range: 5 to 1000 ug/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	90	600	605	5	6.3
B :	90	300	297	-3	5.5
A+B :	44	900	902	2	9.9
A-B :	44	300	307	7	5.9
C :	89	75.0	74.9	-0.1	5.07
D :	90	35.0	32.2	-2.8	2.85
C+D :	44	110.0	108.4	-1.6	7.70
C-D :	44	40.0	43.9	3.9	4.03

s.d(AB): Sw(within run): 4.2 S(between runs): 5.9 S/Sw: 1.41
s.d(CD): Sw(within run): 2.85 S(between runs): 4.11 S/Sw: 1.44

On any given day the calibration is accepted if the values obtained lie within the ranges:

855 to 945 for A+B
270 to 330 for A-B
101.0 to 119.0 for C+D
34.0 to 46.0 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
79	0 - 50	3.2	12.69
22	50 - 100	2.8	3.75
13	100 - 200	7.2	4.79
7	200 - 500	5.7	1.62
0	500 - 1000	N/A	N/A
121	Overall	3.9	N/A

DETECTION CRITERION: 5

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	50	0.3	0.74
Standard Cal :	45	3.9	0.36

NITROGEN - AMMONIA PLUS AMMONIUM
QUALITY CONTROL DATA FROM 03/06/82 TO 22/12/83

LAB: Dorset

Analytical Range: 4 to 1000 ug/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	309	750	749	-1	8.4
B :	309	250	246	-4	5.9
A+B :	225	1000	994	-6	10.5
A-B :	225	500	504	4	9.3
C :	308	75.0	76.1	1.1	2.16
D :	308	25.0	22.8	-2.2	1.77
C+D :	224	100.0	99.3	-0.7	2.91
C-D :	224	50.0	53.4	3.4	2.50

s.d(AB): Sw(within run): 6.6 S(between runs): 7.3 S/Sw: 1.11
s.d(CD): Sw(within run): 1.77 S(between runs): 1.97 S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

955 to 1045 for A+B
470 to 530 for A-B
91.0 to 109.0 for C+D
44.0 to 56.0 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
258	0 - 50	2.1	8.59
67	50 - 100	2.3	3.08
53	100 - 200	2.9	1.90
49	200 - 500	8.5	2.43
23	500 - 1000	6.1	0.81
450	Overall	4.2	N/A

DETECTION CRITERION: 4

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	309	0.1	0.36
Standard Cal :	226	3.9	0.74

NITROGEN - AMMONIA PLUS AMMONIUM
QUALITY CONTROL DATA FROM 04/01/84 TO 20/12/84

LAB: Dorset

Analytical Range: 2 to 1000 ug/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	54	750	750	-0	6.1
B :	54	250	246	-4	5.7
A+B :	54	1000	996	-4	9.1
A-B :	54	500	504	4	7.5
C :	49	75.0	75.2	0.2	1.10
D :	49	25.0	24.6	-0.4	1.95
C+D :	49	100.0	99.8	-0.2	2.45
C-D :	49	50.0	50.6	0.6	2.00

s.d(AB): Sw(within run): 5.3 S(between runs): 5.9 S/Sw: 1.11
s.d(CD): Sw(within run): 1.41 S(between runs): 1.58 S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

955 to 1045 for A+B
470 to 530 for A-B
91.0 to 109.0 for C+D
44.0 to 56.0 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
86	0 - 50	1.5	6.03
12	50 - 100	5.2	6.96
12	100 - 200	3.3	2.23
7	200 - 500	3.9	1.12
8	500 - 1000	3.3	0.43
125	Overall	2.8	N/A

DETECTION CRITERION: 2

OTHER CHECKS:	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	52	0.3	0.53
Standard Cal :	55	3.8	0.49

*** NITROGEN - AMMONIA PLUS AMMONIUM ***

IDENTIFICATION:

Laboratory	: Precipitation	Method Introduced:	01/05/84
LIS Test Name Code:	NNHTFR, NNHTUR	Units	: mg/L as N
Work Station Code	: PRNUT	Unit Code	: 064807
Method Code	: 103CC2	Supervisor	: M. Rawlings
Sample Type/Matrix: Precipitation, Throughfall, Stemflow			

SAMPLING:

Quantity Required: 15 mL
Container : Polystyrene

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance : 1.1 at 5.00 mg/L as N level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm light path at 630 nm.

REPORTING:

Maximum Significant Figures: 3	
Minimum Increment (W) : 0.005	Detection Criterion (T): 0.01

CALIBRATION:

BL plus 4 standards

CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA
Drift : BL plus 3 standards

MODIFICATIONS:

01/05/84- The procedure introduced on this date is the same as Method A for nitrogen-ammonia in HAMES except that the samples are not filtered and the full scale concentration is 5.00 mg/L as N.

NITROGEN - AMMONIA PLUS AMMONIUM

QUALITY CONTROL DATA FROM 14/05/84 TO 20/12/84

LAB: Precipitation

Analytical Range: 0.006 to 5.000 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	50	4.000	4.003	0.003	0.0157
B :	50	0.800	0.802	0.002	0.0100
A+B :	50	4.800	4.805	0.005	0.0182
A-B :	50	3.200	3.201	0.001	0.0189
C :	50	0.800	0.801	0.001	0.0042
D :	50	0.200	0.193	-0.007	0.0090
C+D :	50	1.000	0.994	-0.006	0.0101
C-D :	50	0.600	0.607	0.007	0.0098

s.d(AB): Sw(within run): 0.0134

S(between runs): 0.0131

S/Sw: 0.98

s.d(CD): Sw(within run): 0.0069

S(between runs): 0.0070

S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.575	to	5.025	for A+B
3.050	to	3.350	for A-B
0.955	to	1.045	for C+D
0.570	to	0.630	for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
23	0.000 - 0.100	0.0034	6.89
62	0.100 - 0.500	0.0076	2.54
30	0.500 - 1.000	0.0124	1.66
9	1.000 - 2.000	0.0251	1.67
1	2.000 - 5.000	N/A	N/A
125	Overall	0.0121	N/A

DETECTION CRITERION: 0.006

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Std Cal :	49	1	0.1

*** NITROGEN - AMMONIA PLUS AMMONIUM ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/78
LIS Test Name Code: NNHTFR Units : mg/L as N
Work Station Code : RNDNP Unit Code : 064807
Method Code : 103DC2 Supervisor : J. Crowther
Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance : 0.5 at 2.00 mg/L as N level.

N.B. Nitrate plus nitrite, nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W) : 0.002

Detection Criterion (T): 0.006

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL plus one standard

MODIFICATIONS:

01/02/84 - Sample filtration was eliminated for all sample classes but Great Lakes (G).

15/05/84 - Microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration technique was changed from linear interpolation to the use of a quadratic.

01/10/84 - Sample filtration was eliminated for Great Lakes (G) samples.

NITROGEN - AMMONIA PLUS AMMONIUM
QUALITY CONTROL DATA FROM 04/01/82 TO 30/12/82

LAB: Rivers and Lakes

Analytical Range: 0.006 to 2.00 mg/L as N

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	207	1.500	1.503	0.003	0.0205
B :	208	0.500	0.500	-0.000	0.0101
A+B :	206	2.000	2.003	0.003	0.0262
A-B :	206	1.000	1.004	0.004	0.0190
C :	206	0.300	0.299	-0.001	0.0040
D :	206	0.100	0.098	-0.002	0.0043
C+D :	205	0.400	0.397	-0.003	0.0067
C-D :	205	0.200	0.202	0.002	0.0048

s.d(AB): Sw(within run): 0.0134

S(between runs): 0.0162

S/Sw: 1.20

s.d(CD): Sw(within run): 0.0034

S(between runs): 0.0041

S/Sw: 1.22

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.910	to	2.090	for A+B
0.940	to	1.060	for A-B
0.382	to	0.418	for C+D
0.188	to	0.212	for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
245	0.000 - 0.080	0.0037	9.21
39	0.080 - 0.200	0.0052	3.69
17	0.200 - 0.400	0.0050	1.67
16	0.400 - 1.00	0.009	1.30
8	1.00 - 2.00	0.008	0.56
325	Overall	0.005	N/A

DETECTION CRITERION: 0.006

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Long Term Blank :	202	0.002	0.0015
Standard Cal :	207	281.3	30.40

NITROGEN - AMMONIA PLUS AMMONIUM

QUALITY CONTROL DATA FROM 06/01/83 TO 20/03/84

LAB: Rivers and Lakes

Analytical Range: 0.007 to 2.00 mg/L as N

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	213	1.500	1.512	0.012	0.0219
B :	213	0.500	0.510	0.010	0.0123
A+B :	210	2.000	2.022	0.022	0.0301
A-B :	210	1.000	1.002	0.002	0.0191
C :	222	0.300	0.299	-0.001	0.0051
D :	222	0.100	0.101	0.001	0.0035
C+D :	210	0.400	0.399	-0.001	0.0071
C-D :	210	0.200	0.198	-0.002	0.0050

s.d(AB): Sw(within run): 0.0135 S(between runs): 0.0178 S/Sw: 1.31

s.d(CD): Sw(within run): 0.0035 S(between runs): 0.0044 S/Sw: 1.24

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.910	to	2.090	for A+B
0.940	to	1.060	for A-B
0.382	to	0.418	for C+D
0.188	to	0.212	for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
472	0.000 - 0.080	0.0042	10.46
65	0.080 - 0.200	0.0036	2.56
32	0.200 - 0.400	0.0067	2.22
31	0.400 - 1.00	0.020	2.92
11	1.00 - 2.00	0.020	1.34
611	Overall	0.007	N/A

DETECTION CRITERION: 0.007

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Long Term Blank :	193	0.001	0.0010
Standard Cal :	207	327	21.8

NITROGEN - AMMONIA PLUS AMMONIUM

QUALITY CONTROL DATA FROM 23/08/84 TO 27/12/84

LAB: Rivers and Lakes

Analytical Range: 0.004 to 2.00 mg/L as N

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	58	1.60	1.62	0.02	0.008
B :	58	0.40	0.41	0.01	0.026
A+B :	58	2.00	2.03	0.03	0.027
A-B :	58	1.20	1.21	0.01	0.028
C :	58	0.40	0.41	0.01	0.028
D :	57	0.20	0.20	0.00	0.003
C+D :	57	0.60	0.62	0.02	0.006
C-D :	57	0.20	0.21	0.01	0.005

s.d(AB): Sw(within run): 0.020

S(between runs): 0.019

S/Sw: 0.98

s.d(CD): Sw(within run): 0.003

S(between runs): 0.020

S/Sw: 5.80

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.91	to	2.09	for A+B
1.14	to	1.26	for A-B
0.58	to	0.62	for C+D
0.19	to	0.21	for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
136	0.000 - 0.080	0.0025	6.30
16	0.080 - 0.200	0.0055	3.95
4	0.200 - 0.400	0.0030	1.00
0	0.400 - 1.00	N/A	N/A
1	1.00 - 2.00	N/A	N/A
157	Overall	0.003	N/A

DETECTION CRITERION: 0.004

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Long Term Blank :	58	0.006	0.0041

*** NITROGEN - AMMONIA PLUS AMMONIUM ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/04/77
LIS Test Name Code: NNHTFR Units : mg/L as N
Work Station Code : SNH3P Unit Code : 064807
Method Code : 103AC2 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,
Effluents

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on a filtered sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance : 0.3 at 20.0 mg/L as N level.

N.B. Reactive orthophosphate is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus one 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm.
Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W) : 0.05

Detection Criterion (T): 0.2

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : LTBL plus 4 standards, eg, QCA

Drift : BL plus one standard

NITROGEN - AMMONIA PLUS AMMONIUM

QUALITY CONTROL DATA FROM 07/01/82 TO 22/12/82

LAB: Sewage/Industrial

Analytical Range: 0.1 to 50.0 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	118	35.0	35.0	-0.0	0.40
B :	117	14.0	14.1	0.1	0.18
A+B :	116	49.0	49.0	0.0	0.49
A-B :	116	21.0	20.9	-0.1	0.38
C :	117	14.0	14.1	0.1	0.18
D :	117	3.5	3.5	-0.0	0.07
C+D :	116	17.5	17.6	0.1	0.22
C-D :	116	10.5	10.6	0.1	0.17

s.d(AB): Sw(within run): 0.27

S(between runs): 0.31

S/Sw: 1.15

s.d(CD): Sw(within run): 0.12

S(between runs): 0.14

S/Sw: 1.17

On any given day the calibration is accepted if the values obtained lie within the ranges:

46.8	to	51.3	for A+B
19.5	to	22.5	for A-B
16.6	to	18.4	for C+D
9.9	to	11.1	for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
59	0.0 - 4.0	0.05	2.72
21	4.0 - 10.0	0.13	1.86
43	10.0 - 20.0	0.17	1.13
27	20.0 - 35.0	0.36	1.32
0	35.0 - 50.0	N/A	N/A
150	Overall	0.21	N/A

DETECTION CRITERION: 0.1

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	118	4.76	0.738

NITROGEN - AMMONIA PLUS AMMONIUM

QUALITY CONTROL DATA FROM 05/01/83 TO 13/12/83

LAB: Sewage/Industrial

Analytical Range: 0.2 to 50.0 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	107	35.0	35.1	0.1	0.39
B :	107	14.0	14.0	0.0	0.22
A+B :	106	49.0	49.1	0.1	0.53
A-B :	106	21.0	21.0	0.0	0.35
C :	107	14.0	14.1	0.1	0.18
D :	107	3.5	3.4	-0.1	0.07
C+D :	106	17.5	17.6	0.1	0.22
C-D :	106	10.5	10.7	0.2	0.17

s.d(AB): Sw(within run): 0.25

S(between runs): 0.32

S/Sw: 1.27

s.d(CD): Sw(within run): 0.12

S(between runs): 0.14

S/Sw: 1.16

On any given day the calibration is accepted if the values obtained lie within the ranges:

46.8	to	51.3	for A+B
19.5	to	22.5	for A-B
16.6	to	18.4	for C+D
9.9	to	11.1	for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
82	0.0 - 4.0	0.14	6.85
35	4.0 - 10.0	0.22	3.14
69	10.0 - 20.0	0.18	1.18
47	20.0 - 35.0	0.43	1.57
11	35.0 - 50.0	0.57	1.33
244	Overall	0.30	N/A

DETECTION CRITERION: 0.2

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	107	4.74	0.724

NITROGEN - AMMONIA PLUS AMMONIUM

QUALITY CONTROL DATA FROM 11/01/84 TO 19/12/84

LAB: Sewage/Industrial

Analytical Range: 0.2 to 50.0 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	118	35.0	35.2	0.2	0.51
B :	117	14.0	14.2	0.2	0.24
A+B :	116	49.0	49.4	0.4	0.67
A-B :	116	21.0	21.0	-0.0	0.38
C :	117	14.0	14.1	0.1	0.18
D :	117	3.5	3.4	-0.1	0.24
C+D :	116	17.5	17.5	-0.0	0.30
C-D :	116	10.5	10.7	0.2	0.29

s.d(AB): Sw(within run): 0.27

S(between runs): 0.40

S/Sw: 1.47

s.d(CD): Sw(within run): 0.21

S(between runs): 0.21

S/Sw: 1.01

On any given day the calibration is accepted if the values obtained lie within the ranges:

46.8	to	51.3	for A+B
19.5	to	22.5	for A-B
16.6	to	18.4	for C+D
9.9	to	11.1	for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
153	0.0 - 4.0	0.10	4.80
20	4.0 - 10.0	0.25	3.52
41	10.0 - 20.0	0.38	2.56
14	20.0 - 35.0	0.50	1.80
0	35.0 - 50.0	N/A	N/A
228	Overall	0.28	N/A

DETECTION CRITERION: 0.2

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	108	4.39	0.981

*** NITROGEN - NITRATE ***

IDENTIFICATION:

Laboratory	: Precipitation	Method Introduced:	01/04/78
LIS Test Name Code:	NNO3UR	Units	: mg/L as N
Work Station Code	: PRIC1	Unit Code	: 064807
Method Code	: 003AIO	Supervisor	: M. Rawlings
Sample Type/Matrix: Precipitation, Throughfall, Stemflow.			

SAMPLING:

Quantity Required: 15 mL
Container : Polystyrene

ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by comparison of the sample scan to a series of standard scans.
Full scale conductivity : 10 uS/cm.
Sulphate and chloride are determined simultaneously.

INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.01 Detection Criterion (T): 0.02

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : 1 standard every 10 samples.

NITROGEN - NITRATE

QUALITY CONTROL DATA FROM 06/01/82 TO 24/12/82

LAB: Precipitation

Analytical Range: 0.02 to 2.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	251	1.60	1.60	-0.00	0.017
B :	251	0.40	0.40	-0.00	0.009
A+B :	250	2.00	2.00	-0.00	0.020
A-B :	250	1.20	1.20	0.00	0.019

s.d(AB): Sw(within run): 0.014 S(between runs): 0.014 S/Sw: 1.01

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.91 to 2.09 for A+B
1.14 to 1.26 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
151	0.00 - 0.40	0.010	4.85
118	0.40 - 1.00	0.019	2.75
20	1.00 - 2.00	0.016	1.09
289	Overall	0.016	N/A

DETECTION CRITERION: 0.02

NITROGEN - NITRATE

QUALITY CONTROL DATA FROM 04/01/83 TO 22/12/83

LAB: Precipitation

Analytical Range: 0.02 to 2.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	321	1.60	1.60	-0.00	0.020
B :	320	0.40	0.39	-0.01	0.012
A+B :	319	2.00	1.99	-0.01	0.020
A-B :	319	1.20	1.21	0.01	0.026

s.d(AB): Sw(within run): 0.018

S(between runs): 0.016

S/Sw: 0.89

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.91 to 2.09 for A+B
1.14 to 1.26 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
137	0.00 - 0.40	0.014	6.98
110	0.40 - 1.00	0.013	1.90
24	1.00 - 2.00	0.081	5.38
271	Overall	0.028	N/A

DETECTION CRITERION: 0.02

NITROGEN - NITRATE

QUALITY CONTROL DATA FROM 09/01/84 TO 20/12/84

LAB: Precipitation

Analytical Range: 0.03 to 2.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	168	1.60	1.61	0.01	0.022
B :	168	0.40	0.39	-0.01	0.017
A+B :	167	2.00	2.00	-0.00	0.027
A-B :	167	1.20	1.21	0.01	0.028

s.d(AB): Sw(within run): 0.020 S(between runs): 0.019 S/Sw: 0.97

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.91 to 2.09 for A+B
1.14 to 1.26 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
137	0.00 - 0.40	0.016	7.89
121	0.40 - 1.00	0.022	3.10
43	1.00 - 2.00	0.038	2.56
301	Overall	0.023	N/A

DETECTION CRITERION: 0.03

*** NITROGEN - NITRATE ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/07/80
LIS Test Name Code: NNO3FR NNRICF Units : ug/Filter as N
Work Station Code : PRSEQ Unit Code : 361807
Method Code : 004AI0 Supervisor : M. Rawlings
Sample Type/Matrix: Teflon and nylon filters from sequential filter packs and
nylon filters from LoVol filter packs.

SAMPLING

Quantity Required: 1 filter
Container : Polyethylene bags

SAMPLE PREPARATION:

Filters are extracted with 25.0 mL of DDW (Teflon) or 25.0 mL of 0.03N NaOH
(nylon) in polystyrene tubes with ultrasonic treatment followed by a 24 hour
rest period.

ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample extract by automated
suppressed ion chromatography using an eluent mixture of 0.003M sodium
bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples
are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain
background conductivity. The concentration of nitrate in mg/L as N is determined
by comparison of the sample scan to a series of standard scans. Results are
converted to ug/filter as N.
Full scale conductivity : 30 uS/cm.
Sulphate and chloride are determined simultaneously.

INSTRUMENTATION:

-Ultrasonic bath; polystyrene tubes
-Basic modular continuous flow ion chromatographic system plus microcomputer
for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.25 ug/filter Detection Criterion (T): 0.5 ug/filter

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : 1 standard every 10 samples.

MODIFICATIONS:

01/07/80 - Ion chromatographic procedure for precipitation samples was modified
for analysis of Teflon and nylon filter extracts by developing the above filter
extraction procedure.

10/03/84 - Microcomputer for automated sampling and timing was introduced. At
that time automated spiking of samples with Na₂CO₃/NaHCO₃ was introduced.

NOTES:

Detection criterion is based on duplicate analyses of the extract from one
filter because duplicate filters are not received.

NITROGEN - NITRATE

QUALITY CONTROL DATA FROM 06/01/82 TO 03/12/82

LAB: Precipitation

Analytical Range: 0.48 to 50.0 ug/filter as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	152	40.0	40.0	0.0	0.75
B :	153	10.0	10.3	0.3	0.29
A+B :	151	50.0	50.3	0.3	0.89
A-B :	151	30.0	29.8	-0.2	0.72

s.d(AB): Sw(within run): 0.51 S(between runs): 0.57 S/Sw: 1.13

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.8 to 52.3 for A+B
28.5 to 31.5 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
58	0.00 - 10.00	0.293	5.85
10	10.00 - 25.0	0.34	1.93
3	25.0 - 50.0	0.38	1.02
71	Overall	0.32	N/A

DETECTION CRITERION: 0.48

NITROGEN - NITRATE

QUALITY CONTROL DATA FROM 07/01/83 TO 27/12/83

LAB: Precipitation

Analytical Range: 1.21 to 50.0 ug/filter as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	77	40.0	39.8	-0.2	0.82
B :	77	10.0	10.2	0.2	0.38
A+B :	77	50.0	50.0	-0.0	1.07
A-B :	77	30.0	29.6	-0.4	0.69

s.d(AB): Sw(within run): 0.49

S(between runs): 0.64

S/Sw: 1.30

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.8 to 52.3 for A+B
28.5 to 31.5 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
25	0.00 - 10.00	0.740	14.79
7	10.00 - 25.0	1.69	9.63
2	25.0 - 50.0	1.06	2.83
34	Overall	1.03	N/A

DETECTION CRITERION: 1.21

NITROGEN - NITRATE

QUALITY CONTROL DATA FROM 03/01/84 TO 27/12/84

LAB: Precipitation

Analytical Range: 0.52 to 50.0 ug/filter as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	137	40.0	39.9	-0.1	0.53
B :	138	10.0	10.1	0.1	0.30
A+B :	136	50.0	50.0	0.0	0.65
A-B :	136	30.0	29.9	-0.1	0.57

s.d(AB): Sw(within run): 0.40

S(between runs): 0.43

S/Sw: 1.07

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.8 to 52.3 for A+B
28.5 to 31.5 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
119	0.00 - 10.00	0.319	6.38
19	10.00 - 25.0	0.60	3.42
9	25.0 - 50.0	0.63	1.69
147	Overall	0.43	N/A

DETECTION CRITERION: 0.52

*** NITROGEN - NITRATE ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/07/80
LIS Test Name Code: NND3UR Units : ug/Filter as N
Work Station Code : PRLOV Unit Code : 361807
Method Code : 004AIC Supervisor : M. Rawlings
Sample Type/Matrix: W40 filters from LoVol filter packs.

SAMPLING:

Quantity Required: 1 filter
Container : Polyethylene bag

SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polyethylene tubes with ultrasonic treatment followed by a 24 hour rest period.

ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as N.

Full scale conductivity : 30 uS/cm

N.B. Sulphate and chloride are determined simultaneously.

INSTRUMENTATION:

-Ultrasonic bath; polyethylene tubes
-Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W) : 0.5 ug/filter Detection Criterion (T): 1 ug/filter

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples

MODIFICATIONS:

01/08/81 - Ion chromatographic procedure for precipitation samples was modified for analysis of LoVol W40 filter extracts by developing the above filter extraction procedure.

10/03/84 - Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na₂CO₃/NaHCO₃ was introduced.

NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

NITROGEN - NITRATE

QUALITY CONTROL DATA FROM 06/01/82 TO 03/12/82

LAB: Precipitation

Analytical Range: 0.96 to 100.0 ug/filter as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	152	80.0	80.1	0.1	1.50
B :	153	20.0	20.5	0.5	0.58
A+B :	151	100.0	100.6	0.6	1.78
A-B :	151	60.0	59.5	-0.5	1.43

s.d(AB): Sw(within run): 1.01

S(between runs): 1.14

S/Sw: 1.13

On any given day the calibration is accepted if the values obtained lie within the ranges:

95.5 to 104.5 for A+B
57.0 to 63.0 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
58	0.00 - 20.00	0.585	5.85
10	20.00 - 50.0	0.68	1.93
3	50.0 - 100.0	0.76	1.02
71	Overall	0.64	N/A

DETECTION CRITERION: 0.96

NITROGEN - NITRATE

QUALITY CONTROL DATA FROM 07/01/83 TO 27/12/83

LAB: Precipitation

Analytical Range: 2.43 to 100.0 ug/filter as N

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	77	80.0	79.5	-0.5	1.63
B :	77	20.0	20.4	0.4	0.77
A+B :	77	100.0	99.9	-0.1	2.14
A-B :	77	60.0	59.2	-0.8	1.39

s.d(AB): Sw(within run): 0.98

S(between runs): 1.28

S/Sw: 1.30

On any given day the calibration is accepted if the values obtained lie within the ranges:

95.5 to 104.5 for A+B

57.0 to 63.0 for A-B

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
25	0.00 - 20.00	1.479	14.79
7	20.00 - 50.0	3.37	9.63
2	50.0 - 100.0	2.12	2.83
34	Overall	2.07	N/A

DETECTION CRITERION: 2.43

NITROGEN - NITRATE

QUALITY CONTROL DATA FROM 03/01/84 TO 27/12/84

LAB: Precipitation

Analytical Range: 1.05 to 100.0 ug/filter as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	137	80.0	79.9	-0.1	1.07
B :	138	20.0	20.1	0.1	0.59
A+B :	136	100.0	100.0	0.0	1.30
A-B :	136	60.0	59.8	-0.2	1.14

s.d(AB): Sw(within run): 0.81 S(between runs): 0.86 S/Sw: 1.07

On any given day the calibration is accepted if the values obtained lie within the ranges:

95.5 to 104.5 for A+B
57.0 to 63.0 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
119	0.00 - 20.00	0.638	6.38
19	20.00 - 50.0	1.20	3.42
9	50.0 - 100.0	1.27	1.69
147	Overall	0.85	N/A

DETECTION CRITERION: 1.05

*** NITROGEN - NITRATE PLUS NITRITE ***

IDENTIFICATION:

Laboratory	: Domestic Water	Method Introduced:	01/04/78
LIS Test Name Code:	NNOTUR	Units	: mg/L as N
Work Station Code	: WFN03	Unit Code	: 064807
Method Code	: 002CC2	Supervisor	: M. Rawlings
Sample Type/Matrix:	Ministry of Health Water Samples		

SAMPLING:

Quantity Required: 75 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl)ethylenediaminedihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.
Approximate absorbance : 0.5 at 20.0 mg/L as N level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37 C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 5.0 cm. light path at 520 nm.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.1 Detection Criterion (T): 0.2

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : BL plus 2 standards
Interference: Nitrite standard confirms effective operation of reduction step. Nitrate standards spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirm effective operation of ion exchange column.

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 05/01/82 TO 20/12/82

LAB: Domestic Water

Analytical Range: 0.1 to 20.0 mg/L as N

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	74	15.0	15.0	-0.0	0.18
B :	74	3.0	3.0	0.0	0.14
A+B :	74	18.0	18.0	0.0	0.27
A-B :	74	12.0	11.9	-0.1	0.17

s.d(AB): Sw(within run): 0.12

S(between runs): 0.16

S/Sw: 1.35

On any given day the calibration is accepted if the values obtained lie within the ranges:

17.1 to 18.9 for A+B
11.4 to 12.6 for A-B

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
125	0.0 - 4.0	0.07	3.70
29	4.0 - 10.0	0.12	1.73
14	10.0 - 20.0	0.12	0.78
168	Overall	0.09	N/A

DETECTION CRITERION: 0.1

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 04/01/83 TO 21/12/83

LAB: Domestic Water

Analytical Range: 0.1 to 20.0 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	59	15.0	15.0	0.0	0.17
B :	59	3.0	3.1	0.1	0.06
A+B :	59	18.0	18.1	0.1	0.19
A-B :	59	12.0	12.0	-0.0	0.18

s.d(AB): Sw(within run): 0.13

S(between runs): 0.13

S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

17.1 to 18.9 for A+B
11.4 to 12.6 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
119	0.0 - 4.0	0.04	1.84
24	4.0 - 10.0	0.10	1.49
15	10.0 - 20.0	0.07	0.48
158	Overall	0.06	N/A

DETECTION CRITERION: 0.1

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 19/01/84 TO 21/12/84

LAB: Domestic Water

Analytical Range: 0.1 to 20.0 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	66	15.0	15.0	-0.0	0.13
B :	66	3.0	3.1	0.1	0.08
A+B :	66	18.0	18.0	0.0	0.18
A-B :	66	12.0	11.9	-0.1	0.12

s.d(AB): Sw(within run): 0.09 S(between runs): 0.11 S/Sw: 1.23

On any given day the calibration is accepted if the values obtained lie within the ranges:

17.1 to 18.9 for A+B
11.4 to 12.6 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
149	0.0 - 4.0	0.05	2.40
24	4.0 - 10.0	0.06	0.79
12	10.0 - 20.0	0.09	0.59
185	Overall	0.05	N/A

DETECTION CRITERION: 0.1

*** NITROGEN - NITRATE PLUS NITRITE ***

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 13/06/78
Supervisor : F. Tomassini Units : ug/L as N
Sample Type/Matrix: Streams, Lakes, Precipitation

SAMPLING:

Quantity Required: 50 mL
Container : Plastic (polystyrene)

SAMPLE PREPARATION:

Samples are filtered through 0.45u membrane filters.

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the filtrate of a sample. Nitrate is reduced to nitrite in alkaline media at 37 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl)ethylenediaminedihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance : 0.5 at the 500 ug/L as N level.

N.B. Ammonia plus ammonium is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37 C heating bath (7.7 ml. delay), ion exchange column. Colourimetric measurement is through a 5.0 cm. light path at 520 nm.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W) : 2

Detection Criterion (T): 3

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA

Drift : BL plus 1 standard

Interference: Nitrite standard confirms effective operation of reduction step. Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective operation of ion exchange column.

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 13/01/82 TO 03/06/82

LAB: Dorset

Analytical Range: 4 to 500 ug/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	94	380	379	-1	3.2
B :	94	200	199	-1	11.9
A+B :	47	580	576	-4	8.6
A-B :	47	180	180	0	9.2

s.d(AB): Sw(within run): 6.5

S(between runs): 8.7

S/Sw: 1.34

On any given day the calibration is accepted if the values obtained lie within the ranges:

535 to 625 for A+B
150 to 210 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
31	0 - 50	2.5	10.16
32	50 - 100	2.7	3.64
34	100 - 200	6.6	4.38
21	200 - 300	3.0	1.20
8	300 - 500	2.3	0.58
126	Overall	4.1	N/A

DETECTION CRITERION: 4

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	35	0.0	0.00
Standard Cal :	46	3.12	0.169

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 08/06/82 TO 22/12/83

LAB: Dorset

Analytical Range: 3 to 500 ug/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	208	350	355	5	3.7
B :	208	125	132	7	3.9
A+B :	151	475	487	12	6.4
A-B :	151	225	222	-3	4.1

s.d(AB): Sw(within run): 2.9

S(between runs): 3.8

S/Sw: 1.32

On any given day the calibration is accepted if the values obtained lie within the ranges:

430 to 520 for A+B
195 to 255 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
129	0 - 50	2.1	8.42
83	50 - 100	2.9	3.85
72	100 - 200	2.5	1.70
21	200 - 300	4.9	1.95
16	300 - 500	8.0	1.99
321	Overall	3.3	N/A

DETECTION CRITERION: 3

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	181	0.0	0.24
Standard Cal :	152	3.02	0.176

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 10/01/84 TO 20/12/84

LAB: Dorset

Analytical Range: 5 to 500 ug/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	49	350	352	2	2.4
B :	49	125	130	5	3.8
A+B :	49	475	482	7	5.4
A-B :	49	225	222	-3	3.3

s.d(AB): Sw(within run): 2.4 S(between runs): 3.2 S/Sw: 1.35

On any given day the calibration is accepted if the values obtained lie within the ranges:

430 to 520 for A+B
195 to 255 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
57	0 - 50	2.8	11.10
26	50 - 100	5.4	7.19
21	100 - 200	2.8	1.86
4	200 - 300	7.1	2.83
12	300 - 500	6.2	1.54
120	Overall	4.5	N/A

DETECTION CRITERION: 5

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	45	0.0	0.00
Standard Cal :	49	2.96	0.202

*** NITROGEN - NITRATE PLUS NITRITE ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/78
LIS Test Name Code: NNOTFR Units : mg/L as N
Work Station Code : RNDNP Unit Code : 064807
Method Code : 102DC2 Supervisor : J. Crowther
Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl)ethylenediaminedihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance : 0.5 at the 5.00 mg/L as N level.

N.B. Ammonia plus ammonium, nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37 C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 1.5 cm. light path at 520 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W) : 0.005

Detection Criterion (T): 0.02

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration : LTBL plus 3 standards

Drift : BL plus 1 standard

Interference: Nitrite standard confirms effective operation of reduction step. Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective operation of ion exchange column.

MODIFICATIONS:

01/02/84 - Sample filtration was eliminated for all sample classes but Great Lakes (G).

15/05/84 - Microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration technique was changed from linear interpolation to the use of a quadratic.

01/10/84 - Sample filtration was eliminated for Great Lakes (G) samples.

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 01/01/82 TO 30/12/82

LAB: Rivers and Lakes

Analytical Range: 0.015 to 5.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	195	3.75	3.77	0.02	0.069
B :	197	1.25	1.24	-0.01	0.034
A+B :	194	5.00	5.01	0.01	0.088
A-B :	194	2.50	2.53	0.03	0.064
C :	198	0.750	0.755	0.005	0.0096
D :	195	0.250	0.242	-0.008	0.0068
C+D :	194	1.000	0.997	-0.003	0.0130
C-D :	194	0.500	0.513	0.013	0.0105

s.d(AB): Sw(within run): 0.046 S(between runs): 0.054 S/Sw: 1.19
s.d(CD): Sw(within run): 0.0075 S(between runs): 0.0083 S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.78 to 5.23 for A+B
2.35 to 2.65 for A-B
0.955 to 1.045 for C+D
0.470 to 0.530 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
130	0.000 - 0.200	0.0094	9.37
88	0.200 - 0.500	0.0104	2.97
35	0.500 - 1.000	0.0560	7.47
56	1.000 - 2.50	0.036	2.06
31	2.50 - 5.00	0.086	2.29
340	Overall	0.039	N/A

DETECTION CRITERION: 0.015

OTHER CHECKS:	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	195	0.003	0.0024
Standard Cal :	198	209.8	34.50

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 06/01/83 TO 20/03/84

LAB: Rivers and Lakes

Analytical Range: 0.019 to 5.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	206	3.75	3.78	0.03	0.070
B :	202	1.25	1.23	-0.02	0.025
A+B :	193	5.00	5.02	0.02	0.088
A-B :	193	2.50	2.55	0.05	0.058
C :	208	0.750	0.750	0.000	0.0086
D :	207	0.250	0.237	-0.013	0.0070
C+D :	195	1.000	0.987	-0.013	0.0130
C-D :	195	0.500	0.513	0.013	0.0086

s.d(AB): Sw(within run): 0.041 S(between runs): 0.053 S/Sw: 1.29
s.d(CD): Sw(within run): 0.0061 S(between runs): 0.0078 S/Sw: 1.29

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.78 to 5.23 for A+B
2.35 to 2.65 for A-B
0.955 to 1.045 for C+D
0.470 to 0.530 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
221	0.000 - 0.200	0.0119	11.87
106	0.200 - 0.500	0.0115	3.28
66	0.500 - 1.000	0.0237	3.16
111	1.000 - 2.50	0.044	2.49
63	2.50 - 5.00	0.102	2.72
567	Overall	0.044	N/A

DETECTION CRITERION: 0.019

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	178	0.002	0.0025
Standard Cal :	189	246	31.7

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 23/08/84 TO 27/12/84

LAB: Rivers and Lakes

Analytical Range: 0.021 to 5.00 mg/L as N

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	60	4.00	3.99	-0.01	0.034
B :	60	1.00	1.00	0.00	0.016
A+B :	60	5.00	4.99	-0.01	0.038
A-B :	60	3.00	2.98	-0.02	0.037
C :	60	1.000	1.002	0.002	0.0160
D :	60	0.500	0.508	0.008	0.0145
C+D :	60	1.500	1.510	0.010	0.0268
C-D :	60	0.500	0.494	-0.006	0.0145

s.d(AB): Sw(within run): 0.026

S(between runs): 0.026

S/Sw: 1.02

s.d(CD): Sw(within run): 0.0103

S(between runs): 0.0152

S/Sw: 1.48

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.78 to 5.23 for A+B
2.85 to 3.15 for A-B
1.455 to 1.545 for C+D
0.470 to 0.530 for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
75	0.000 - 0.200	0.0129	12.88
28	0.200 - 0.500	0.0131	3.75
21	0.500 - 1.000	0.0342	4.55
23	1.000 - 2.50	0.039	2.25
12	2.50 - 5.00	0.056	1.48
159	Overall	0.030	N/A

DETECTION CRITERION: 0.021

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Long Term Blank :	60	0.013	0.0097

*** NITROGEN - NITRATE PLUS NITRITE ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/04/76
LIS Test Name Code: NNOTFR Units : mg/L as N
Work Station Code : SNO2NO3 Unit Code : 064807
Method Code : 102CC2 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,
Effluents

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N(1-naphthyl)ethylenediaminedihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance : 0.2 at 20.0 mg/L as N level.

N.B. Nitrite is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37 C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 1.5 cm light path at 520 nm. Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W) : 0.05

Detection Criterion (T): 0.1

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : LTBL plus 4 standards

Drift : BL plus 1 standard

Interference: Nitrite standard confirms effective operation of reduction step. Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective operation of ion exchange column.

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 05/01/82 TO 22/12/82

LAB: Sewage/Industrial

Analytical Range: 0.1 to 50.0 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	124	35.0	36.2	1.2	0.34
B :	125	15.4	15.7	0.3	0.48
A+B :	123	50.4	51.9	1.5	0.58
A-B :	123	19.6	20.5	0.9	0.60
C :	125	15.4	15.4	0.0	0.24
D :	125	4.2	4.0	-0.2	0.08
C+D :	124	19.6	19.4	-0.2	0.27
C-D :	124	11.2	11.4	0.2	0.23

s.d(AB): Sw(within run): 0.43
s.d(CD): Sw(within run): 0.16

S(between runs): 0.42
S(between runs): 0.18

S/Sw: 0.99
S/Sw: 1.10

On any given day the calibration is accepted if the values obtained lie within the ranges:

48.2 to 52.7 for A+B
18.1 to 21.1 for A-B
18.7 to 20.5 for C+D
10.6 to 11.8 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
213	0.0 - 4.0	0.05	2.41
33	4.0 - 10.0	0.13	1.83
21	10.0 - 20.0	0.15	0.97
15	20.0 - 35.0	0.24	0.86
0	35.0 - 50.0	N/A	N/A
282	Overall	0.10	N/A

DETECTION CRITERION: 0.1

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	125	6.00	0.844

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 18/02/83 TO 13/12/83

LAB: Sewage/Industrial

Analytical Range: 0.1 to 50.0 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	83	35.0	36.2	1.2	0.52
B :	80	15.4	16.4	1.0	0.31
A+B :	80	50.4	52.6	2.2	0.69
A-B :	80	19.6	19.8	0.2	0.42
C :	83	15.4	15.5	0.1	0.18
D :	83	4.2	4.0	-0.2	0.09
C+D :	83	19.6	19.5	-0.1	0.24
C-D :	83	11.2	11.5	0.3	0.16

s.d(AB): Sw(within run): 0.29

S(between runs): 0.42

S/Sw: 1.44

s.d(CD): Sw(within run): 0.11

S(between runs): 0.14

S/Sw: 1.31

On any given day the calibration is accepted if the values obtained lie within the ranges:

48.2	to	52.7	for A+B
18.1	to	21.1	for A-B
18.7	to	20.5	for C+D
10.6	to	11.8	for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
62	0.0 - 4.0	0.04	1.97
1	4.0 - 10.0	N/A	N/A
4	10.0 - 20.0	0.08	0.54
1	20.0 - 35.0	N/A	N/A
0	35.0 - 50.0	N/A	N/A
68	Overall	0.10	N/A

DETECTION CRITERION: 0.1

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	80	6.50	0.794

NITROGEN - NITRATE PLUS NITRITE

QUALITY CONTROL DATA FROM 05/01/84 TO 20/12/84

LAB: Sewage/Industrial

Analytical Range: 0.4 to 50.0 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	104	35.0	35.6	0.6	0.62
B :	104	15.4	15.9	0.5	0.57
A+B :	103	50.4	51.5	1.1	1.11
A-B :	103	19.6	19.7	0.1	0.43
C :	104	15.4	15.5	0.1	0.16
D :	104	4.2	4.2	-0.0	0.16
C+D :	103	19.6	19.7	0.1	0.25
C-D :	103	11.2	11.4	0.2	0.19

s.d(AB): Sw(within run): 0.31

S(between runs): 0.59

S/Sw: 1.95

s.d(CD): Sw(within run): 0.13

S(between runs): 0.16

S/Sw: 1.22

On any given day the calibration is accepted if the values obtained lie within the ranges:

48.2	to	52.7	for A+B
18.1	to	21.1	for A-B
18.7	to	20.5	for C+D
10.6	to	11.8	for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
157	0.0 - 4.0	0.23	11.61
37	4.0 - 10.0	0.18	2.60
20	10.0 - 20.0	0.24	1.60
4	20.0 - 35.0	0.81	2.94
1	35.0 - 50.0	N/A	N/A
219	Overall	0.25	N/A

DETECTION CRITERION: 0.4

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	99	6.92	0.850

*** NITROGEN - NITRITE ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/78
LIS Test Name Code: NNO2FR Units : mg/L as N
Work Station Code : RNDNP Unit Code : 064807
Method Code : 102DC2 Supervisor : J. Crowther
Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide and N(1-naphthyl)-ethylenediamine dihydrochloride. Approximate absorbance : 0.6 at the 0.25 mg/L as N level.
N.B. Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm.
Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.0005 Detection Criterion (T): 0.002

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration : LTBL plus 3 standards, e.g., QCA
Drift : BL plus 1 standard

MODIFICATIONS:

01/02/84 - Sample filtration was eliminated for all sample classes but Great Lakes (G).
15/05/84 - Microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration technique was changed from linear interpolation to the use of a quadratic.
01/10/84 - Sample filtration was eliminated for Great Lakes (G) samples.

NITROGEN - NITRITE

QUALITY CONTROL DATA FROM 04/01/82 TO 30/12/82

LAB: Rivers and Lakes

Analytical Range: 0.003 to 0.100 mg/L as N

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
C :	199	0.0750	0.0758	0.0008	0.00109
D :	199	0.0250	0.0251	0.0001	0.00067
C+D :	198	0.1000	0.1008	0.0008	0.00146
C-D :	198	0.0500	0.0507	0.0007	0.00107

s.d(CD): Sw(within run): 0.00076 S(between runs): 0.00090 S/Sw: 1.20

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.0955 to 0.1045 for C+D
0.0470 to 0.0530 for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
255	0.000 - 0.020	0.0016	15.54
42	0.020 - 0.050	0.0021	5.88
28	0.050 - 0.100	0.0023	3.08
325	Overall	0.0017	N/A

DETECTION CRITERION: 0.003

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Long Term Blank :	198	0.0006	0.00050
Standard Cal :	198	441.3	21.35

NITROGEN - NITRITE

QUALITY CONTROL DATA FROM 06/01/83 TO 20/03/84

LAB: Rivers and Lakes

Analytical Range: 0.006 to 0.100 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
C :	206	0.0750	0.0748	-0.0002	0.00396
D :	206	0.0250	0.0247	-0.0003	0.00065
C+D :	194	0.1000	0.0996	-0.0004	0.00418
C-D :	194	0.0500	0.0501	0.0001	0.00406

s.d(CD): Sw(within run): 0.00287 S(between runs): 0.00284 S/Sw: 0.99

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.0910 to 0.1090 for C+D
0.0440 to 0.0560 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
435	0.000 - 0.020	0.0035	34.91
69	0.020 - 0.050	0.0041	11.66
39	0.050 - 0.100	0.0021	2.84
543	Overall	0.0035	N/A

DETECTION CRITERION: 0.006

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	180	0.0004	0.00037
Standard Cal :	188	389	33.1

NITROGEN - NITRITE

QUALITY CONTROL DATA FROM 30/05/84 TO 27/12/84

LAB: Rivers and Lakes

Analytical Range: 0.001 to 0.25 mg/L as N

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	101	0.2000	0.2004	0.0004	0.00219
B :	101	0.0500	0.0502	0.0002	0.00132
A+B :	100	0.2500	0.2505	0.0005	0.00267
A-B :	100	0.1500	0.1502	0.0002	0.00236
C :	101	0.0500	0.0500	-0.0000	0.00274
D :	101	0.0250	0.0251	0.0001	0.00079
C+D :	100	0.0750	0.0751	0.0001	0.00299
C-D :	100	0.0250	0.0248	-0.0002	0.00264

s.d(AB): Sw(within run): 0.00167 S(between runs): 0.00181 S/Sw: 1.08
s.d(CD): Sw(within run): 0.00187 S(between runs): 0.00202 S/Sw: 1.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.2410 to 0.2590 for A+B
0.1440 to 0.1560 for A-B
0.0660 to 0.0840 for C+D
0.0190 to 0.0310 for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
211	0.000 - 0.020	0.0008	8.40
28	0.020 - 0.050	0.0014	3.86
14	0.050 - 0.100	0.0017	2.23
13	0.100 - 0.25	0.007	4.00
266	Overall	0.002	N/A

DETECTION CRITERION: 0.001

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Long Term Blank :	101	0.0014	0.00250

*** NITROGEN - NITRITE ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/04/76
LIS Test Name Code: NNO2FR Units : mg/L as N
Work Station Code : SNO2NO3 Unit Code : 064807
Method Code : 102CC2 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,
Effluents

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide and N(1-naphthyl)-ethylenediamine dihydrochloride. Approximate absorbance : 0.4 at the 2.0 mg/L as N level.
N.B. Nitrate plus nitrite is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.005 Detection Criterion (T): 0.03

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA
Drift : BL plus one standard

NITROGEN - NITRITE

QUALITY CONTROL DATA FROM 07/01/82 TO 22/12/82

LAB: Sewage/Industrial

Analytical Range: 0.01 to 2.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
C :	107	1.40	1.42	0.02	0.013
D :	107	0.70	0.70	0.00	0.016
C+D :	106	2.10	2.12	0.02	0.025
C-D :	106	0.70	0.71	0.01	0.015

s.d(CD): Sw(within run): 0.010

S(between runs): 0.015

S/Sw: 1.39

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.01 to 2.19 for C+D
0.64 to 0.76 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
238	0.00 - 0.40	0.005	2.33
6	0.40 - 1.00	0.009	1.26
1	1.00 - 2.00	N/A	N/A
245	Overall	0.005	N/A

DETECTION CRITERION: 0.01

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	107	4.16	0.359

NITROGEN - NITRITE

QUALITY CONTROL DATA FROM 05/01/83 TO 13/12/83

LAB: Sewage/Industrial

Analytical Range: 0.02 to 2.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
C :	93	1.40	1.42	0.02	0.031
D :	91	0.70	0.70	0.00	0.023
C+D :	91	2.10	2.12	0.02	0.042
C-D :	91	0.70	0.71	0.01	0.032

s.d(CD): Sw(within run): 0.023 S(between runs): 0.027 S/Sw: 1.20

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.01 to 2.19 for C+D
0.64 to 0.76 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
95	0.00 - 0.40	0.011	5.65
5	0.40 - 1.00	0.013	1.86
2	1.00 - 2.00	0.014	0.94
102	Overall	0.012	N/A

DETECTION CRITERION: 0.02

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	95	4.21	0.407

NITROGEN - NITRITE

QUALITY CONTROL DATA FROM 05/01/84 TO 20/12/84

LAB: Sewage/Industrial

Analytical Range: 0.03 to 2.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
C :	100	1.40	1.39	-0.01	0.031
D :	100	0.70	0.69	-0.01	0.026
C+D :	100	2.10	2.07	-0.03	0.047
C-D :	100	0.70	0.70	0.00	0.032

s.d(CD): Sw(within run): 0.023 S(between runs): 0.028 S/Sw: 1.26

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.01 to 2.19 for C+D
0.64 to 0.76 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
154	0.00 - 0.40	0.018	8.86
18	0.40 - 1.00	0.114	16.28
10	1.00 - 2.00	0.065	4.31
182	Overall	0.044	N/A

DETECTION CRITERION: 0.03

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	97	4.40	0.387

*** NITROGEN - TOTAL KJELDAHL ***

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 07/10/80
Supervisor : F. Tomassini Units : ug/L as N
Sample Type/Matrix: Streams, Lakes, Precipitation

SAMPLING:

Quantity Required: 50 mL
Container : Plastic (polystyrene)

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters kept at 200 C and 360 C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst. Approximate absorbance : 0.25 at the 1000 ug/L as N level.

INSTRUMENTATION:

-Block digesters(2)
-Basic automated modular continuous flow system plus 1 module:37 C bath(7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 5 Detection Criterion (T): 15

CALIBRATION:

BL plus 2 undigested standards

CONTROLS:

Calibration : LTBL plus 2 undigested standards, eg, QCA
Recovery : 3 digested B1 plus 3 digested standards in duplicate, eg, R1
Drift : BL plus 1 undigested standard

MODIFICATIONS

04/06/84- Analytical procedure suspended.

NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

NITROGEN - TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 12/01/82 TO 17/12/82

LAB: Dorset

Analytical Range: 7 to 1000 ug/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	168	750	749	-1	3.9
B :	168	250	250	-0	2.8
A+B :	167	1000	998	-2	5.6
A-B :	167	500	499	-1	4.0

s.d(AB): Sw(within run): 2.8

S(between runs): 3.4

S/Sw: 1.21

On any given day the calibration is accepted if the values obtained lie within the ranges:

978 to 1023 for A+B
485 to 515 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	336	720	705	20.5
R2 :	336	420	425	16.3
R3 :	336	140	142	8.9

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
0	0 - 50	N/A	N/A
16	50 - 100	4.6	6.07
211	100 - 200	12.1	8.06
212	200 - 500	25.8	7.38
20	500 - 1000	30.7	4.10
459	Overall	20.5	N/A

DETECTION CRITERION: 7

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	163	-1	2.2
Digested Blank :	168	-4	7.2
Standard Cal :	168	6.92	1.069

NITROGEN - TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 05/01/83 TO 22/12/83

LAB: Dorset

Analytical Range: 14 to 1000 ug/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	122	750	750	0	4.9
B :	122	250	251	1	2.5
A+B :	121	1000	1001	1	6.3
A-B :	121	500	500	-0	4.4

s.d(AB): Sw(within run): 3.1

S(between runs): 3.9

S/Sw: 1.23

On any given day the calibration is accepted if the values obtained lie within the ranges:

978 to 1023 for A+B
485 to 515 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	122	720	707	13.0
R2 :	113	420	425	9.1
R3 :	122	140	141	4.3

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
1	0 - 50	N/A	N/A
20	50 - 100	8.6	11.50
171	100 - 200	13.7	9.12
140	200 - 500	16.5	4.70
8	500 - 1000	9.2	1.22
340	Overall	14.7	N/A

DETECTION CRITERION: 14

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	122	-0	3.5
Digested Blank :	122	-7	6.6
Standard Cal :	122	7.43	0.431

NITROGEN - TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 06/01/84 TO 01/06/84

LAB: Dorset

Analytical Range: 16 to 1000 ug/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	41	750	749	-1	3.3
B :	41	250	255	5	3.6
A+B :	41	1000	1004	4	6.1
A-B :	41	500	495	-5	3.3

s.d(AB): Sw(within run): 2.3

S(between runs): 3.5

S/Sw: 1.48

On any given day the calibration is accepted if the values obtained lie within the ranges:

978 to 1023 for A+B
485 to 515 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	43	720	694	13.6
R2 :	44	420	144	5.7

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
1	0 - 50	N/A	N/A
4	50 - 100	10.0	13.33
25	100 - 200	8.1	5.39
78	200 - 500	20.5	5.85
12	500 - 1000	17.2	2.30
120	Overall	18.0	N/A

DETECTION CRITERION: 16

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	42	3	4.5
Digested Blank :	41	6	9.8
Standard Cal :	43	7.26	0.464

*** NITROGEN-TOTAL KJELDAHL ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/79
LIS Test Name Code: NNTKUR Units : mg/L as N
Work Station Code : RTNP Unit Code : 064807
Method Code : 004AC2 Supervisor : J. Crowther
Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters kept at 200 C and 360 C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst.
Approximate absorbance : 0.5 at the 2.0 mg/L as N level.
N.B. Total phosphorus is determined simultaneously.

INSTRUMENTATION:

- Block digesters(2)
- Basic automated modular continuous flow system plus 1 module:37 C bath(7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm.
- Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.01 Detection Criterion (T): 0.04

CALIBRATION:

BL plus 4 undigested standards

CONTROLS:

Calibration : LTBL plus 2 undigested standards, eg, QCA
Recovery : 3 digested BL plus 3 digested standards in duplicate, eg, R1
Drift : BL plus 1 undigested standard

MODIFICATIONS:

15/08/83 - Microcomputer system was introduced. At this time the calibration technique was changed from linear interpolation to the use of a quadratic.

NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

NITROGEN - TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 05/01/82 TO 23/12/82

LAB: Rivers and Lakes

Analytical Range: 0.04 to 2.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	154	1.500	1.499	-0.001	0.0121
B :	154	0.500	0.497	-0.003	0.0065
A+B :	153	2.000	1.996	-0.004	0.0152
A-B :	153	1.000	1.002	0.002	0.0123

s.d(AB): Sw(within run): 0.0087 S(between runs): 0.0097 S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.910 to 2.090 for A+B
0.940 to 1.060 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	292	1.40	1.41	0.023
R2 :	299	0.84	0.84	0.021
R3 :	304	0.28	0.28	0.013

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
341	0.00 - 0.40	0.021	10.71
131	0.40 - 1.00	0.019	2.67
20	1.00 - 2.00	0.034	2.27
492	Overall	0.021	N/A

DETECTION CRITERION: 0.04

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	151	0.005	0.0050
Digested Blank :	254	0.021	0.0163
Standard Cal :	153	282.7	43.27

NITROGEN - TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 05/01/83 TO 05/12/83

LAB: Rivers and Lakes

Analytical Range: 0.05 to 2.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	143	1.500	1.503	0.003	0.0118
B :	143	0.500	0.503	0.003	0.0114
A+B :	142	2.000	2.006	0.006	0.0197
A-B :	142	1.000	0.999	-0.001	0.0122

s.d(AB): Sw(within run): 0.0087 S(between runs): 0.0116 S/Sw: 1.34

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.910 to 2.090 for A+B
0.940 to 1.060 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	267	1.40	1.36	0.047
R2 :	268	0.84	0.82	0.035
R3 :	267	0.28	0.28	0.019

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
188	0.00 - 0.40	0.032	15.99
105	0.40 - 1.00	0.032	4.50
11	1.00 - 2.00	0.044	2.96
304	Overall	0.033	N/A

DETECTION CRITERION: 0.05

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	145	0.007	0.0072
Digested Blank :	364	0.019	0.0138
Standard Cal :	72	347	36.1

NITROGEN - TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 03/01/84 TO 31/12/84

LAB: Rivers and Lakes

Analytical Range: 0.03 to 2.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	155	1.500	1.494	-0.006	0.0108
B :	155	0.500	0.507	0.007	0.0149
A+B :	154	2.000	2.000	0.000	0.0206
A-B :	154	1.000	0.987	-0.013	0.0159

s.d(AB): Sw(within run): 0.0113 S(between runs): 0.0130 S/Sw: 1.16

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.910 to 2.090 for A+B
0.940 to 1.060 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	307	1.40	1.38	0.041
R2 :	307	0.84	0.83	0.027
R3 :	310	0.28	0.28	0.031

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
188	0.00 - 0.40	0.021	10.33
110	0.40 - 1.00	0.035	4.93
28	1.00 - 2.00	0.036	2.41
326	Overall	0.028	N/A

DETECTION CRITERION: 0.03

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	155	0.011	0.0036
Digested Blank :	155	0.018	0.0101
Standard Cal :	57	419	34.4

*** NITROGEN - TOTAL KJELDAHL ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/04/79
LIS Test Name Code: NNTKUR Units : mg/L as N
Work Station Code : STKNP Unit Code : 064807
Method Code : 504BC2 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Domestic Waters, Effluents,
Leachates

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters kept at 200 C and 360 C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst. Approximate absorbance : 0.8 at the 10.0 mg/L as N level.
N.B. Total phosphorus is determined simultaneously.

INSTRUMENTATION:

-Block digesters(2)
-Basic automated modular continuous flow system plus 1 module:37 C bath(7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.05 Detection Criterion (T): 0.3

CALIBRATION:

BL plus 1 undigested standard

CONTROLS:

Calibration : LTBL plus 2 undigested standards, eg, QCA
Recovery : 2 digested BL plus 2 digested standards in duplicate, eg, R1
Drift : BL plus 1 undigested standard

NITROGEN - TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 05/01/82 TO 29/12/82

LAB: Sewage/Industrial

Analytical Range: 0.07 to 10.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	145	7.00	7.03	0.03	0.088
B :	80	1.40	1.43	0.03	0.050
A+B :	79	8.40	8.47	0.07	0.132
A-B :	79	5.60	5.61	0.01	0.097

s.d(AB): Sw(within run): 0.069

S(between runs): 0.072

S/Sw: 1.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

7.95 to 8.85 for A+B
5.30 to 5.90 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	134	7.00	6.99	0.120
R2 :	135	3.50	3.52	0.066

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
46	0.00 - 2.00	0.042	4.15
59	2.00 - 5.00	0.071	2.03
46	5.00 - 10.00	0.071	0.94
151	Overall	0.068	N/A

DETECTION CRITERION: 0.07

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	155	1.30	0.331

NITROGEN - TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 07/01/83 TO 22/12/83

LAB: Sewage/Industrial

Analytical Range: 0.17 to 10.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	117	7.00	7.08	0.08	0.119
B :	120	1.40	1.47	0.07	0.062
A+B :	116	8.40	8.56	0.16	0.159
A-B :	116	5.60	5.61	0.01	0.104

s.d(AB): Sw(within run): 0.073 S(between runs): 0.095 S/Sw: 1.29

On any given day the calibration is accepted if the values obtained lie within the ranges:

7.95 to 8.85 for A+B
5.30 to 5.90 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	116	7.00	6.99	0.187
R2 :	115	3.50	3.54	0.105

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
57	0.00 - 2.00	0.101	10.11
39	2.00 - 5.00	0.098	2.80
31	5.00 - 10.00	0.145	1.94
127	Overall	0.117	N/A

DETECTION CRITERION: 0.17

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	121	1.38	0.497

NITROGEN - TOTAL KJELDAHL

QUALITY CONTROL DATA FROM 05/01/84 TO 28/12/84

LAB: Sewage/Industrial

Analytical Range: 0.16 to 10.00 mg/L as N

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	138	7.00	7.08	0.08	0.079
B :	138	1.40	1.48	0.08	0.044
A+B :	137	8.40	8.56	0.16	0.101
A-B :	137	5.60	5.60	-0.00	0.079

s.d(AB): Sw(within run): 0.056 S(between runs): 0.064 S/Sw: 1.14

On any given day the calibration is accepted if the values obtained lie within the ranges:

7.95 to 8.85 for A+B
5.30 to 5.90 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	124	7.00	6.97	0.120
R2 :	124	3.50	3.52	0.094

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
187	0.00 - 2.00	0.099	9.89
48	2.00 - 5.00	0.146	4.16
31	5.00 - 10.00	0.191	2.55
266	Overall	0.131	N/A

DETECTION CRITERION: 0.16

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	130	1.83	0.559

*** OXYGEN-BIOCHEMICAL DEMAND ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74
LIS Test Name Code: BOD5 Units : mg/L as O
Work Station Code : R5DBOD Unit Code : 064808
Method Code : 101A12 Supervisor : J. Crowther
Sample Type/Matrix: Rivers, Lakes, Effluents

SAMPLING:

Quantity Required: 400 mL
Container : Glass or plastic

SAMPLE PREPARATION:

If necessary sample pH is adjusted to neutral and chlorine is removed by reaction with sodium sulphite.

ANALYTICAL PROCEDURE:

Using dissolved oxygen (DO) analyses, samples are measured for oxygen depletion after a five day period (BOD5) of storage in the dark at 20 C. If necessary dilutions are made with aerated, nutrient-enriched water to obtain a 50-75% oxygen depletion. If the sample has undergone any of the sample preparation steps listed above or if the sample is an industrial waste, a sewage seed is added. For such samples calculation of an appropriate seed correction is required.

INSTRUMENTATION:

- Weston and Stack Oxygen analyzer plus DO probe equipped with stirrer and fitted with a Teflon membrane of 0.5 mil thickness which is permeable to oxygen.
- Titration equipment for Winkler analysis of dissolved oxygen
- Incubator (19-21 C); BOD bottles (300mL)

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.01 Detection Criterion (T): 0.5

CALIBRATION(DO):

Blank is a sulphite solution(negligible DO) and the standard is air-saturated distilled water. The DO content of the latter is read from a table after measuring its temperature and the barometric pressure in the laboratory.

CONTROLS:

Calibration(DO): 2 "solutions" of distilled water which have been partially stripped of DO by flushing with nitrogen. These "solutions", of different but unknown DO, are analyzed with the DO probe and by the Winkler titration procedure. The difference between the values for the two analytical methods is utilized as a slope control for the DO probe.

Recovery(BOD5): 2 BL plus 3 standards, eg, R1; the expected BOD5 is 67% of the oxygen requirement for complete oxidation.

Drift(DO) : Air saturated distilled water after every 20 samples.

MODIFICATIONS

01/07/82 -Quality control program for DO was expanded, and the use of standard 300 mL BOD bottles was restored.

OXYGEN DEMAND - BIOCHEMICAL

QUALITY CONTROL DATA FROM 06/08/82 TO 31/12/82

LAB: Rivers and Lakes

Analytical Range: 0.3 to 20.0 mg/L as O

CALIBRATION CONTROL:

		<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A	:	39	0.00	0.12	0.12	0.147
B	:	39	0.00	0.19	0.19	0.154

On any given day the calibration is accepted if the values obtained for A and B lie within the range:

-0.45 to 0.45

RECOVERIES:

		<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1	:	21	4.34	3.87	0.688
R2	:	22	2.17	2.16	0.563

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
63	0.0 - 2.0	0.16	16.01
8	2.0 - 5.0	0.63	17.88
0	5.0 - 10.0	N/A	N/A
0	10.0 - 20.0	N/A	N/A
71	Overall	0.27	N/A

DETECTION CRITERION: 0.3

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
5 day DDW Blank :	22	0.24	0.200
5 day BOD Blank :	21	0.27	0.230

OXYGEN DEMAND - BIOCHEMICAL

QUALITY CONTROL DATA FROM 05/01/83 TO 30/12/83

LAB: Rivers and Lakes

Analytical Range: 0.3 to 20.0 mg/L as O

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	103	0.00	0.16	0.16	0.212
B :	101	0.00	0.23	0.23	0.193

On any given day the calibration is accepted if the values obtained for A and B lie within the range:

-0.45 to 0.45

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	53	4.34	4.30	0.364
R2 :	51	2.17	2.21	0.342

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
165	0.0 - 2.0	0.20	19.75
14	2.0 - 5.0	0.26	7.44
1	5.0 - 10.0	N/A	N/A
0	10.0 - 20.0	N/A	N/A
180	Overall	0.20	N/A

DETECTION CRITERION: 0.3

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
5 day DDW Blank :	47	0.30	0.117
5 day BOD Blank :	48	0.27	0.158

OXYGEN DEMAND - BIOCHEMICAL

QUALITY CONTROL DATA FROM 04/01/84 TO 12/12/84

LAB: Rivers and Lakes

Analytical Range: 0.3 to 20.0 mg/L as O

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	100	0.00	0.15	0.15	0.137
B :	100	0.00	0.17	0.17	0.097

On any given day the calibration is accepted if the values obtained for A and B lie within the range:

-0.45 to 0.45

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	52	4.34	4.29	0.577
R2 :	51	2.17	2.20	0.239

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
136	0.0 - 2.0	0.16	15.65
10	2.0 - 5.0	0.07	2.03
1	5.0 - 10.0	N/A	N/A
0	10.0 - 20.0	N/A	N/A
147	Overall	0.15	N/A

DETECTION CRITERION: 0.3

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
5 day DDW Blank :	54	0.22	0.169
5 day BOD Blank :	54	0.24	0.202

*** OXYGEN - BIOCHEMICAL DEMAND ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: Before '61
LIS Test Name Code: BOD5 Units : mg/L as O
Work Station Code : SBBOD5 Unit Code : 064808
Method Code : 101A12 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Effluents, Domestic Waters,
Leachates

SAMPLING:

Quantity Required: 400 mL
Container : Glass or plastic

SAMPLE PREPARATION:

If necessary sample pH is adjusted to neutral and chlorine is removed by reaction with sodium sulphite.

ANALYTICAL PROCEDURE:

Using dissolved oxygen (DO) analyses, samples are measured for oxygen depletion after a five day period (BOD5) of storage in the dark at 20C. If necessary dilutions are made with aerated, nutrient-enriched water to obtain a 50-75% oxygen depletion. If the sample has undergone any of the sample preparation steps listed above or if the sample is an industrial waste, a sewage seed is added. For such samples calculation of an appropriate seed correction is required.

INSTRUMENTATION:

- Weston and Stack Oxygen analyzer plus DO probe equipped with stirrer and fitted with a Teflon membrane of 0.5 mil thickness which is permeable to oxygen.
- Titration equipment for Winkler analysis of dissolved oxygen
- Incubator (19-21 C); BOD bottles (300mL)

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.01 Detection Criterion (T): 3.6

CALIBRATION(DO):

Blank is a sulphite solution(negligible DO) and the standard is air-saturated distilled, deionized water. The DO content of the latter is read from a table after measuring its temperature and the barometric pressure in the laboratory.

CONTROLS:

Calibration(DO): 2 "solutions" of distilled water which have been partially stripped of DO by flushing with nitrogen. These "solutions", of different but unknown DO, are analyzed with the DO probe and by the Winkler titration procedure. The difference between the values for the two analytical methods is utilized as a slope control for the DO probe.

Recovery(BOD5): 2 BL plus 3 standards, eg, R1; the expected BOD5 is 67% of the oxygen requirement for complete oxidation.

Drift(DO) : Air saturated distilled water after every 24 samples.

MODIFICATIONS

01/05/81 -Quality control program for DO was expanded, and the use of standard 300 mL BOD bottles was restored.

OXYGEN DEMAND - BIOCHEMICAL

QUALITY CONTROL DATA FROM 22/04/82 TO 22/12/82

LAB: Sewage/Industrial

Analytical Range: 2 to 400 mg/L as O

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	114	0.00	0.11	0.11	0.085
B :	113	0.00	0.09	0.09	0.082

On any given day the calibration is accepted if the values obtained for A and B lie within the range:

-0.25 to 0.25

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	56	2.17	2.18	0.124
R2 :	57	4.34	4.21	0.470
R3 :	52	6.52	6.41	0.268

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
28	0 - 20	1.2	12.00
20	20 - 50	2.3	6.66
30	50 - 100	5.1	6.80
32	100 - 400	5.9	2.34
110	Overall	37.7	N/A

DETECTION CRITERION: 2

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
5 day DDW Blank :	57	0.16	0.111
5 day BOD Blank :	52	0.12	0.127

OXYGEN DEMAND - BIOCHEMICAL

QUALITY CONTROL DATA FROM 04/01/83 TO 30/12/83

LAB: Sewage/Industrial

Analytical Range: 2 to 400 mg/L as O

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	119	0.00	0.11	0.11	0.090
B :	120	0.00	0.08	0.08	0.066

On any given day the calibration is accepted if the values obtained for A and B lie within the range:

-0.25 to 0.25

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	61	2.17	2.15	0.196
R2 :	63	4.34	4.31	0.253
R3 :	63	6.52	6.42	0.368

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
22	0 - 20	1.2	11.84
19	20 - 50	4.3	12.36
32	50 - 100	5.4	7.15
44	100 - 400	16.4	6.57
117	Overall	11.8	N/A

DETECTION CRITERION: 2

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
5 day DDW Blank :	70	0.14	0.086
5 day BOD Blank :	67	0.16	0.133

OXYGEN DEMAND - BIOCHEMICAL

QUALITY CONTROL DATA FROM 04/01/84 TO 24/12/84

LAB: Sewage/Industrial

Analytical Range: 3 to 400 mg/L as O

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	182	0.00	0.05	0.05	0.079
B :	183	0.00	0.06	0.06	0.067

On any given day the calibration is accepted if the values obtained for A and B lie within the range:

-0.25 to 0.25

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	91	2.17	2.09	0.139
R2 :	91	4.34	4.18	0.207
R3 :	91	6.52	6.33	0.263

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
23	0 - 20	2.0	20.22
28	20 - 50	4.7	13.49
51	50 - 100	9.8	13.11
60	100 - 400	11.0	4.39
162	Overall	9.7	N/A

DETECTION CRITERION: 3

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
5 day DDW Blank :	95	0.18	0.094
5 day BOD Blank :	92	0.20	0.111

*** OXYGEN - CHEMICAL DEMAND ***

IDENTIFICATION:

Laboratory	: Rivers and Lakes	Method Introduced:	01/07/82
LIS Test Name Code:	COD	Units	: mg/L as O
Work Station Code	: RCOD	Unit Code	: 064808
Method Code	: 002AC0	Supervisor	: J. Crowther
Sample Type/Matrix:	Rivers, Lakes, Effluents		

SAMPLING:

Quantity Required: 25 mL
Container : Glass

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 150 C. Analysis is completed by automated colourimetric measurement of trivalent chromium.

Approximate absorbance: 0.6 at the 500 mg/L level.

INSTRUMENTATION:

Culture tubes with Teflon closures; mechanical-convection oven
-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 1 Detection Criterion (T): 8

CALIBRATION:

2 digested BL plus 3 digested standards

CONTROLS:

Calibration : 2 digested standards, eg, QCA
Recovery : 2 digested standards, eg, R1
Drift : Undigested BL: 1 per 9 samples
Interference: Digested standard spiked with 300 mg/L Cl confirms suppression of chloride interference.

MODIFICATIONS:

30/06/82- Manual COD procedure described in HAMES was discontinued. Development report on the current procedure, described above, is available on request.

NOTES:

-In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week.
-Chemical oxygen demand analyses for Rivers and Lakes' samples are set up in the latter laboratory, but completed in the Sewage/Industrial laboratory. Thus the following performance data report only refers to duplicate results. Please consult the Sewage/Industrial report for the remaining QC data.

OXYGEN DEMAND - CHEMICAL

QUALITY CONTROL DATA FROM 15/06/82 TO 29/10/84

LAB: Rivers and Lakes

Analytical Range: 8 to 500 mg/L as O

DUPLICATES: (1982)

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
5	0.0 - 10.0	2.23	44.57
37	10.0 - 25.0	2.04	11.64
10	25.0 - 50.0	2.28	6.08
3	50.0 - 200	2.6	2.07
2	200 - 500	7.1	2.02
57	Overall	2.3	N/A

DETECTION CRITERION: 3.7

DUPLICATES: (1983)

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
26	0.0 - 10.0	4.70	93.96
40	10.0 - 25.0	3.79	21.64
7	25.0 - 50.0	4.05	10.80
5	50.0 - 200	8.6	6.91
1	200 - 500	N/A	N/A
79	Overall	4.8	N/A

DETECTION CRITERION: 7.7

DUPLICATES: (1984)

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
12	0.0 - 10.0	1.96	39.14
18	10.0 - 25.0	3.58	20.45
11	25.0 - 50.0	5.55	14.81
7	50.0 - 200	11.1	8.87
1	200 - 500	N/A	N/A
49	Overall	6.6	N/A

DETECTION CRITERION: 3.2

*** OXYGEN - CHEMICAL DEMAND ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/07/82
LIS Test Name Code: COD Units : mg/L as O
Work Station Code : SBCOD Unit Code : 064808
Method Code : 002AC0 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Domestic Waters, Leachates,
Effluents

SAMPLING:

Quantity Required: 25 mL
Container : Glass

ANALYTICAL PROCEDURE:

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 150 C. Analysis is completed by automated colourimetric measurement of trivalent chromium.

Approximate absorbance: 0.6 at the 500 mg/L level.

INSTRUMENTATION:

Culture tubes with Teflon closures; mechanical-convection oven
-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 1 Detection Criterion (T): 5

CALIBRATION:

2 digested BL plus 3 digested standards

CONTROLS:

Calibration : 2 digested standards, eg, QCA
Recovery : 2 digested standards, eg, R1
Drift : 1 undigested BL per 9 samples
Interference: Digested standard spiked with 300 mg/mL Cl confirms suppression of chloride interference.

MODIFICATIONS:

30/06/82- Manual COD procedure described in HAMES was discontinued. Development report on the current procedure, described above, is available on request.

NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis are scheduled for completion within the week.

OXYGEN DEMAND - CHEMICAL

QUALITY CONTROL DATA FROM 03/03/82 TO 21/12/82

LAB: Sewage/Industrial

Analytical Range: 3 to 600 mg/L as O

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	93	400.0	398.3	-1.7	3.73
B :	93	100.0	101.8	1.8	2.22
A+B :	93	500.0	500.1	0.1	4.18
A-B :	93	300.0	296.5	-3.5	4.50

s.d(AB): Sw(within run): 3.18

S(between runs): 3.07

S/Sw: 0.97

On any given day the calibration is accepted if the values obtained lie within the ranges:

477.5 to 522.5 for A+B
285.0 to 315.0 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	92	400	383	5.7
R2 :	91	100	97	3.8

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
42	0 - 20	2.0	19.75
88	20 - 100	2.2	3.69
53	100 - 300	3.8	1.90
16	300 - 500	6.1	1.52
5	500 - 600	7.8	1.42
204	Overall	3.7	N/A

DETECTION CRITERION: 3

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Digested Blank :	89	33.0	4.63
Chloride Check :	81	54.7	14.39
Standard Cal :	94	2.2	0.00

OXYGEN DEMAND - CHEMICAL

QUALITY CONTROL DATA FROM 04/01/83 TO 20/12/83

LAB: Sewage/Industrial

Analytical Range: 4 to 600 mg/L as O

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	90	400.0	397.8	-2.2	5.25
B :	92	100.0	100.3	0.3	3.87
A+B :	90	500.0	498.2	-1.8	7.70
A-B :	90	300.0	297.3	-2.7	5.01

s.d(AB): Sw(within run): 3.54

S(between runs): 4.61

S/Sw: 1.30

On any given day the calibration is accepted if the values obtained lie within the ranges:

477.5 to 522.5 for A+B
285.0 to 315.0 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	88	400	384	8.7
R2 :	92	100	97	5.6

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
8	0 - 20	2.7	26.93
61	20 - 100	5.3	8.76
26	100 - 300	8.0	4.02
12	300 - 500	14.9	3.74
0	500 - 600	N/A	N/A
107	Overall	7.8	N/A

DETECTION CRITERION: 4

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Digested Blank :	91	42.0	7.91
Chloride Check :	88	54.5	5.48
Standard Cal :	92	2.2	0.00

OXYGEN DEMAND - CHEMICAL

QUALITY CONTROL DATA FROM 09/01/84 TO 31/12/84

LAB: Sewage/Industrial

Analytical Range: 11 to 600 mg/L as O

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	65	400.0	399.9	-0.1	8.04
B :	64	100.0	103.3	3.3	5.74
A+B :	64	500.0	503.5	3.5	8.76
A-B :	64	300.0	296.9	-3.1	10.32

s.d(AB): Sw(within run): 7.30

S(between runs): 6.99

S/Sw: 0.96

On any given day the calibration is accepted if the values obtained lie within the ranges:

477.5 to 522.5 for A+B
285.0 to 315.0 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	62	400	398	15.2
R2 :	64	100	98	6.5

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
39	0 - 20	6.6	66.31
51	20 - 100	8.6	14.33
16	100 - 300	17.1	8.57
5	300 - 500	62.0	15.49
0	500 - 600	N/A	N/A
111	Overall	16.3	N/A

DETECTION CRITERION: 11

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Digested Blank :	62	45.6	15.31
Chloride Check :	62	49.7	8.25
Standard Cal :	66	2.2	0.01

*** PH ***

IDENTIFICATION:

Laboratory	: Domestic Water	Method Introduced:	Before '60
LIS Test Name Code:	PH	Units	: Dimensionless
Work Station Code	: WPC	Unit Code	: Nil
Method Code	: 001AI1	Supervisor	: M. Rawlings
Sample Type/Matrix: Domestic Waters, Leachates, Effluents			

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample at room temperature. Stirring rate, and room temperature range are uniform for all samples and standards.

INSTRUMENTATION:

pH meter, stirrer, glass electrode

REPORTING:

Maximum Significant Figures:	3	Detection Criterion (T):	N/A
Minimum Increment (W) :	0.01		

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9.

CONTROLS:

Calibration : 2 buffers

PH

QUALITY CONTROL DATA FROM 04/01/82 TO 21/12/82

LAB: Domestic Water

Analytical Range:0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av.Concn Measured</u>	<u>Av.Bias</u>	<u>Standard Deviation</u>
A :	197	9.00	8.99	-0.01	0.024
B :	196	4.00	4.00	0.00	0.020
A+B :	195	13.00	13.00	-0.00	0.033
A-B :	195	5.00	4.99	-0.01	0.029

s.d(AB): Sw(within run): 0.021 S(between runs): 0.022 S/Sw: 1.06

On any given day the calibration is accepted if the values obtained lie within the ranges:

12.82 to 13.18 for A+B
4.88 to 5.12 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
5	1.00 - 5.00	0.033	1.09
4	5.00 - 6.00	0.091	1.66
32	6.00 - 7.00	0.134	2.06
277	7.00 - 8.00	0.089	1.18
144	8.00 - 12.00	0.043	0.43
462	Overall	0.082	N/A

PH

QUALITY CONTROL DATA FROM 11/01/83 TO 21/12/83

LAB: Domestic Water

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	151	9.00	8.98	-0.02	0.030
B :	151	4.00	4.00	-0.00	0.015
A+B :	150	13.00	12.98	-0.02	0.039
A-B :	150	5.00	4.98	-0.02	0.029

s.d(AB): Sw(within run): 0.020 S(between runs): 0.024 S/Sw: 1.18

On any given day the calibration is accepted if the values obtained lie within the ranges:

12.82 to 13.18 for A+B
4.88 to 5.12 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
2	1.00 - 5.00	0.007	0.24
3	5.00 - 6.00	0.000	0.00
35	6.00 - 7.00	0.410	6.30
213	7.00 - 8.00	0.074	0.98
94	8.00 - 12.00	0.055	0.55
347	Overall	0.149	N/A

PH

QUALITY CONTROL DATA FROM 02/01/84 TO 18/12/84

LAB: Domestic Water

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	155	8.50	8.52	0.02	0.029
B :	155	6.20	6.19	-0.01	0.027
A+B :	153	14.70	14.71	0.01	0.045
A-B :	153	2.30	2.34	0.04	0.029

s.d(AB): Sw(within run): 0.021

S(between runs): 0.028

S/Sw: 1.35

On any given day the calibration is accepted if the values obtained lie within the ranges:

14.52 to 14.88 for A+B
2.18 to 2.42 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
2	1.00 - 5.00	0.007	0.24
8	5.00 - 6.00	0.015	0.27
42	6.00 - 7.00	0.038	0.59
268	7.00 - 8.00	0.039	0.52
86	8.00 - 12.00	0.029	0.29
406	Overall	0.037	N/A

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 01/06/76
Supervisor : F. Tomassini
Sample Type/Matrix: Streams, Lakes, Precipitation

SAMPLING:

Quantity Required: 250 mL
Container : Polyethylene bottle filled to the brim; screw caps with
cone-shaped liners

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (100 mL) at room temperature.
Stirring rate, tube size, degree of electrode immersion, and room temperature
range are uniform for all samples and standards.
N.B. Alkalinity (Gran) or Acidity (Total Fixed Endpoint) is determined
simultaneously.

INSTRUMENTATION

Semi-automated modular titration system with microcomputer control and data
reduction software.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.01

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

CONTROLS:

Calibration: LTBL plus two standards, eg, QCA
Drift : 2 standard buffers -4 times daily

PH

QUALITY CONTROL DATA FROM 05/01/82 TO 09/12/82

LAB: Dorset

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	159	9.18	9.19	0.01	0.024
B :	159	6.86	6.88	0.02	0.010
A+B :	158	16.04	16.08	0.04	0.031
A-B :	158	2.32	2.31	-0.01	0.019

s.d(AB): Sw(within run): 0.013 S(between runs): 0.018 S/Sw: 1.36

On any given day the calibration is accepted if the values obtained lie within the ranges:

15.89 to 16.19 for A+B
2.22 to 2.42 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
0	1.00 - 3.00	N/A	N/A
2	3.00 - 4.00	0.007	0.20
34	4.00 - 5.00	0.009	0.20
115	5.00 - 6.00	0.016	0.29
112	6.00 - 9.00	0.013	0.17
263	Overall	0.014	N/A

PH

QUALITY CONTROL DATA FROM 10/01/84 TO 11/12/84

LAB: Dorset

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	113	6.86	6.87	0.01	0.028
B :	99	4.00	4.01	0.01	0.014
A+B :	99	10.86	10.87	0.01	0.030
A-B :	99	2.86	2.86	0.00	0.034

s.d(AB): Sw(within run): 0.024 S(between runs): 0.022 S/Sw: 0.92

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.71 to 11.01 for A+B
2.76 to 2.96 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
0	1.00 - 3.00	N/A	N/A
3	3.00 - 4.00	0.006	0.16
22	4.00 - 5.00	0.040	0.89
77	5.00 - 6.00	0.113	2.05
63	6.00 - 9.00	0.021	0.28
165	Overall	0.079	N/A

*** PH ***

IDENTIFICATION:

Laboratory	: Precipitation	Method Introduced:	01/05/79
LIS Test Name Code:	PH	Units	: Dimensionless
Work Station Code	: PHACD	Unit Code	: Nil
Method Code	: 002A11	Supervisor	: M. Rawlings
Sample Type/Matrix: Precipitation, Throughfall, Stemflow			

SAMPLING:

Quantity Required: 15 mL
Container : Polystyrene

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards.
N.B. Gran and total fixed endpoint acidity are determined simultaneously.

INSTRUMENTATION:

Automated modular titration system with microcomputer control and data reduction software.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.01

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

CONTROLS:

Calibration : LTBL plus two standards, eg, QCA

MODIFICATIONS:

01/04/82- Sample volume was decreased from 100.0 to 10.0 mL.
01/05/83- System was fully automated by introduction of a sampler, and an automated device for washing the electrode between analyses.

PH

QUALITY CONTROL DATA FROM 03/01/82 TO 23/12/82

LAB: Precipitation

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	210	6.86	6.86	-0.00	0.007
B :	210	4.01	4.01	0.00	0.013
A+B :	209	10.87	10.87	0.00	0.015
A-B :	209	2.85	2.85	-0.00	0.014

s.d(AB): Sw(within run): 0.010 S(between runs): 0.010 S/Sw: 1.04

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.82 to 10.91 for A+B
2.82 to 2.88 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
1	1.00 - 3.00	N/A	N/A
31	3.00 - 4.00	0.040	1.16
93	4.00 - 5.00	0.026	0.58
36	5.00 - 6.00	0.054	0.98
30	6.00 - 9.00	0.081	1.08
191	Overall	0.049	N/A

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Slope :	200	99.2	0.67

PH

QUALITY CONTROL DATA FROM 05/01/83 TO 22/12/83

LAB: Precipitation

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	134	6.86	6.84	-0.02	0.015
B :	134	4.01	4.00	-0.01	0.010
A+B :	133	10.87	10.85	-0.02	0.020
A-B :	133	2.85	2.84	-0.01	0.016

s.d(AB): Sw(within run): 0.011 S(between runs): 0.013 S/Sw: 1.15

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.82 to 10.91 for A+B
2.82 to 2.88 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
0	1.00 - 3.00	N/A	N/A
9	3.00 - 4.00	0.014	0.40
17	4.00 - 5.00	0.038	0.85
6	5.00 - 6.00	0.070	1.28
4	6.00 - 9.00	0.037	0.49
36	Overall	0.046	N/A

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Slope :	53	99.8	0.47

PH

QUALITY CONTROL DATA FROM 03/01/84 TO 21/12/84

LAB: Precipitation

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	162	6.86	6.86	-0.00	0.010
B :	162	4.01	4.01	0.00	0.008
A+B :	161	10.87	10.87	-0.00	0.015
A-B :	161	2.85	2.85	-0.00	0.010

s.d(AB): Sw(within run): 0.007 S(between runs): 0.009 S/Sw: 1.25

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.82 to 10.91 for A+B
2.82 to 2.88 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
1	1.00 - 3.00	N/A	N/A
29	3.00 - 4.00	0.029	0.84
120	4.00 - 5.00	0.121	2.69
20	5.00 - 6.00	0.104	1.90
20	6.00 - 9.00	0.100	1.33
190	Overall	0.109	N/A

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Slope :	159	98.5	0.50

*** PH ***

IDENTIFICATION:

Laboratory	: Rivers and Lakes	Method Introduced:	09/07/80
LIS Test Name Code:	PH	Units	: Dimensionless
Work Station Code	: RMGALK	Unit Code	: Nil
Method Code	: 003AI2	Supervisor	: J. Crowther
Sample Type/Matrix:	Rivers, Lakes		

SAMPLING:

Quantity Required: 150 mL
Container : Polyethylene bottle filled to the brim; screw caps with cone shaped liners

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (100 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards.
N.B. Alkalinity (Gran and total fixed endpoint) are determined simultaneously

INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data reduction software.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.01

CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

CONTROLS:

Calibration: LTBL plus two standard sodium carbonate solutions, eg, QCA
Drift : 2 standard buffers -4 times daily

MODIFICATIONS:

02/03/84- QC program at this workstation was expanded to include pH and total fixed endpoint alkalinity. Preparation and storage of QC solutions was modified. As shown by the following QCA-B data, the attempt to check pH calibrations by monitoring the pH values of the dilute alkalinity standards failed. Buffers will be utilized in '85.

16/03/84- Use of 4 oz. polyethylene bottles plus screw caps with cone-shaped liners was recommended for sampling.

PH

QUALITY CONTROL DATA FROM 02/03/84 TO 28/11/84

LAB: Rivers and Lakes

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	81	N/A	9.76	N/A	0.125
B :	80	N/A	8.30	N/A	0.887
A+B :	80	N/A	18.06	N/A	0.930
A-B :	80	N/A	1.47	N/A	0.860

s.d(AB): Sw(within run): 0.608

S(between runs): 0.633

S/Sw: 1.04

On any given day the calibration is accepted if the values obtained lie within the ranges:

17.79 to 18.33 for A+B
1.28 to 1.64 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
1	1.00 - 5.00	N/A	N/A
11	5.00 - 6.00	0.032	0.59
30	6.00 - 7.00	0.026	0.40
11	7.00 - 8.00	0.011	0.15
0	8.00 - 12.00	N/A	N/A
53	Overall	0.027	N/A

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	81	5.65	0.142

*** PH ***

IDENTIFICATION:

Laboratory	: Rivers and Lakes	Method Introduced:	01/04/74
LIS Test Name Code:	PH	Units	: Dimensionless
Work Station Code	: ROPH	Unit Code	: Nil
Method Code	: 001AI1	Supervisor	: J. Crowther
Sample Type/Matrix:	Rivers, Lakes, Effluents, Soil Extracts		

SAMPLING:

Quantity Required: 75 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (50 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards.

INSTRUMENTATION:

- pH meter, stirrer, glass electrode

REPORTING:

Maximum Significant Figures:	3	
Minimum Increment (W) :	0.01	Detection Criterion (T): N/A

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9.

CONTROLS:

Calibration: 2 standard buffers
Drift : 2 standard buffers -4 times daily

PH

QUALITY CONTROL DATA FROM 04/01/82 TO 23/12/82

LAB: Rivers and Lakes

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	164	8.00	8.01	0.01	0.036
B :	164	6.86	6.87	0.01	0.022
A+B :	163	14.86	14.88	0.02	0.041
A-B :	163	1.14	1.14	-0.00	0.042

s.d(AB): Sw(within run): 0.030 S(between runs): 0.030 S/Sw: 0.99

On any given day the calibration is accepted if the values obtained lie within the ranges:

14.41 to 15.31 for A+B
0.84 to 1.44 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
16	1.00 - 5.00	0.067	2.22
52	5.00 - 7.00	0.042	0.69
170	7.00 - 8.00	0.040	0.53
137	8.00 - 9.00	0.020	0.24
7	9.00 - 12.00	0.021	0.20
382	Overall	0.036	N/A

PH

QUALITY CONTROL DATA FROM 05/01/83 TO 22/12/83

LAB: Rivers and Lakes

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	184	8.00	8.01	0.01	0.048
B :	185	6.86	6.87	0.01	0.011
A+B :	183	14.86	14.87	0.01	0.050
A-B :	183	1.14	1.14	-0.00	0.049

s.d(AB): Sw(within run): 0.035 S(between runs): 0.035 S/Sw: 1.01

On any given day the calibration is accepted if the values obtained lie within the ranges:

14.41 to 15.31 for A+B
0.84 to 1.44 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
23	1.00 - 5.00	0.031	1.04
92	5.00 - 7.00	0.036	0.60
166	7.00 - 8.00	0.086	1.15
153	8.00 - 9.00	0.022	0.26
7	9.00 - 12.00	0.050	0.48
441	Overall	0.058	N/A

PH

QUALITY CONTROL DATA FROM 03/01/84 TO 14/12/84

LAB: Rivers and Lakes

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	171	8.00	8.00	-0.00	0.036
B :	171	6.86	6.88	0.02	0.019
A+B :	170	14.86	14.88	0.02	0.041
A-B :	170	1.14	1.12	-0.02	0.041

s.d(AB): Sw(within run): 0.029

S(between runs): 0.029

S/Sw: 1.00

On any given day the calibration is accepted if the values obtained lie within the ranges:

14.41 to 15.31 for A+B
0.84 to 1.44 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
16	1.00 - 5.00	0.023	0.77
55	5.00 - 7.00	0.065	1.08
168	7.00 - 8.00	0.103	1.37
156	8.00 - 9.00	0.035	0.42
9	9.00 - 12.00	0.034	0.32
404	Overall	0.075	N/A

*** PH ***

IDENTIFICATION:

Laboratory	: Sewage/Industrial	Method Introduced:	Before '70
LIS Test Name Code:	PH	Units	: Dimensionless
Work Station Code	: SBPH	Unit Code	: Nil
Method Code	: 001AI1	Supervisor	: P. Campbell
Sample Type/Matrix:	Sewage, Industrial Waste, Effluents		

SAMPLING:

Quantity Required: 75 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (50 mL) at room temperature. Stirring rate and room temperature range are uniform for all samples and standards.

INSTRUMENTATION:

pH meter, stirrer, glass electrode

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.01

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9.

CONTROLS:

Calibration: 2 standard buffers

PH

QUALITY CONTROL DATA FROM 04/01/82 TO 21/12/82

LAB: Sewage/Industrial

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	96	9.00	8.98	-0.02	0.020
B :	93	4.00	4.02	0.02	0.039
A+B :	92	13.00	13.00	-0.00	0.044
A-B :	92	5.00	4.96	-0.04	0.041

s.d(AB): Sw(within run): 0.029 S(between runs): 0.031 S/Sw: 1.06

On any given day the calibration is accepted if the values obtained lie within the ranges:

12.78 to 13.23 for A+B
4.85 to 5.15 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
7	1.00 - 5.00	0.008	0.25
26	5.00 - 7.00	0.014	0.23
70	7.00 - 8.00	0.017	0.23
18	8.00 - 9.00	0.015	0.17
4	9.00 - 12.00	0.006	0.05
125	Overall	0.016	N/A

PH

QUALITY CONTROL DATA FROM 05/01/83 TO 23/12/83

LAB: Sewage/Industrial

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	122	9.00	8.97	-0.03	0.042
B :	122	4.00	4.01	0.01	0.014
A+B :	121	13.00	12.97	-0.03	0.048
A-B :	121	5.00	4.96	-0.04	0.040

s.d(AB): Sw(within run): 0.028 S(between runs): 0.031 S/Sw: 1.10

On any given day the calibration is accepted if the values obtained lie within the ranges:

12.78 to 13.23 for A+B
4.85 to 5.15 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
6	1.00 - 5.00	0.023	0.77
24	5.00 - 7.00	0.065	1.08
77	7.00 - 8.00	0.017	0.23
12	8.00 - 9.00	0.017	0.20
5	9.00 - 12.00	0.009	0.09
124	Overall	0.032	N/A

PH

QUALITY CONTROL DATA FROM 04/01/84 TO 20/12/84

LAB: Sewage/Industrial

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	118	9.00	8.99	-0.01	0.047
B :	118	4.00	4.00	0.00	0.017
A+B :	117	13.00	12.99	-0.01	0.054
A-B :	117	5.00	4.99	-0.01	0.047

s.d(AB): Sw(within run): 0.033 S(between runs): 0.036 S/Sw: 1.07

On any given day the calibration is accepted if the values obtained lie within the ranges:

12.78 to 13.23 for A+B
4.85 to 5.15 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
8	1.00 - 5.00	0.009	0.31
30	5.00 - 7.00	0.052	0.86
78	7.00 - 8.00	0.062	0.83
17	8.00 - 9.00	0.014	0.17
1	9.00 - 12.00	N/A	N/A
139	Overall	0.589	N/A

*** PHENOLICS - REACTIVE ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74
LIS Test Name Code: PHNOL Units : ug/L as Phenol
Work Station Code : ROPHEN Unit Code : 063704
Method Code : 002BC2 Supervisor : J. Crowther
Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,
Domestic Water Supplies, Leachates, Sewages, Industrial Wastes

SAMPLING:

Quantity Required: 250 mL
Container : Glass
Preservative : Copper sulphate-phosphoric acid
Other : Special bottle containing preservative is available

ANALYTICAL PROCEDURE:

Samples are automatically distilled from an acid media, and reactive phenolics in the distillate are determined colourimetrically by formation of an antipyrene dye through reactions with 4-aminoantipyrene and potassium ferricyanide. Approximate absorbance: 0.03 at the 50 ug/L as phenol level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 505 nm.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.2 Detection Criterion (T): 0.6

CALIBRATION:

BL plus 1 standard

CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA
Drift : BL plus 1 standard

NOTES:

A report identifying reactive phenolics is available on request.

PHENOLICS - REACTIVE

QUALITY CONTROL DATA FROM 05/01/82 TO 23/12/82

LAB: Rivers and Lakes

Analytical Range: 0.9 to 50.0 ug/L as Phenol

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	118	40.0	40.0	0.0	0.65
B :	119	10.0	10.4	0.4	0.33
A+B :	117	50.0	50.4	0.4	0.81
A-B :	117	30.0	29.6	-0.4	0.59

s.d(AB): Sw(within run): 0.42 S(between runs): 0.51 S/Sw: 1.22

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.8 to 52.3 for A+B
28.5 to 31.5 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
230	0.0 - 10.0	0.54	10.82
12	10.0 - 25.0	3.45	19.73
2	25.0 - 50.0	0.71	1.89
244	Overall	1.04	N/A

DETECTION CRITERION: 0.9

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	100	0.4	0.29
Standard Cal :	90	852	20.6

PHENOLICS - REACTIVE

QUALITY CONTROL DATA FROM 04/01/83 TO 07/12/83

LAB: Rivers and Lakes

Analytical Range: 0.6 to 50.0 ug/L as Phenol

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	92	40.0	39.8	-0.2	0.55
B :	92	10.0	10.3	0.3	0.33
A+B :	92	50.0	50.1	0.1	0.77
A-B :	92	30.0	29.5	-0.5	0.46

s.d(AB): Sw(within run): 0.33

S(between runs): 0.45

S/Sw: 1.38

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.8 to 52.3 for A+B
28.5 to 31.5 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
202	0.0 - 10.0	0.34	6.73
9	10.0 - 25.0	1.32	7.56
0	25.0 - 50.0	N/A	N/A
211	Overall	0.47	N/A

DETECTION CRITERION: 0.6

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	90	0.2	0.09
Standard Cal :	92	850	14.3

PHENOLICS - REACTIVE

QUALITY CONTROL DATA FROM 03/01/84 TO 11/12/84

LAB: Rivers and Lakes

Analytical Range: 0.4 to 50.0 ug/L as Phenol

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	99	40.0	39.9	-0.1	0.37
B :	99	10.0	10.3	0.3	0.39
A+B :	99	50.0	50.1	0.1	0.58
A-B :	99	30.0	29.6	-0.4	0.49

s.d(AB): Sw(within run): 0.34

S(between runs): 0.38

S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.8 to 52.3 for A+B
28.5 to 31.5 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
212	0.0 - 10.0	0.27	5.33
14	10.0 - 25.0	0.36	2.07
7	25.0 - 50.0	0.28	0.75
233	Overall	0.28	N/A

DETECTION CRITERION: 0.4

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	104	0.6	2.63
Standard Cal :	99	866	22.9

*** PHOSPHORUS - FILTERED TOTAL ***

IDENTIFICATION:

Laboratory	: Rivers and Lakes	Method Introduced:	01/09/79
LIS Test Name Code:	PPFT	Units	: ug/L as P
Work Station Code	: RNFTP	Unit Code	: 063815
Method Code	: 605AC2	Supervisor	: J. Crowther
Sample Type/Matrix:	Rivers, Lakes, Precipitation		

SAMPLING:

Quantity Required: 35 mL
Container : Specially marked Pyrex culture tubes with Teflon-lined caps
Other : Samples must be field filtered using Sartorius C.A. 0.45u membrane filters, and filtrate must fill designated culture tubes.

ANALYTICAL PROCEDURE:

After withdrawal of excess volume, digestion reagent is added and samples are autoclaved in sulphuric acid-potassium persulphate media at 121 C for 60 min. The orthophosphate content of the digestate is determined colourimetrically by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance : 0.4 at the 200 ug/L as P level.

INSTRUMENTATION:

Autoclave plus basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube. Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.1

Detection Criterion (T): 1.2

CALIBRATION:

BL plus 2 undigested standards

CONTROLS:

Calibration : LTBL plus 4 undigested standards, eg, QCA
Recovery : 3 digested BL plus 4 digested standards, eg, R1
Drift : BL plus 2 undigested standards

MODIFICATIONS:

01/02/84 - Method was terminated in the River laboratory due to limited workload.

NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

PHOSPHORUS - FILTERED TOTAL

QUALITY CONTROL DATA FROM 06/01/82 TO 08/12/82

LAB: Rivers and Lakes

Analytical Range: 0.6 to 200 ug/L as P

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	26	150.0	150.2	0.2	1.02
B :	25	50.0	48.2	-1.8	1.76
A+B :	25	200.0	198.4	-1.6	1.96
A-B :	25	100.0	102.0	2.0	2.12
C :	27	15.0	15.2	0.2	0.22
D :	27	5.0	5.0	-0.0	0.17
C+D :	27	20.0	20.1	0.1	0.30
C-D :	27	10.0	10.2	0.2	0.25

s.d(AB): Sw(within run): 1.50

S(between runs): 1.44

S/Sw: 0.96

s.d(CD): Sw(within run): 0.17

S(between runs): 0.20

S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

191.0	to	209.0	for A+B
94.0	to	106.0	for A-B
18.7	to	21.4	for C+D
9.1	to	10.9	for C-D

RECOVERIES:

	Number of Data	Expected Concn	Av. Concn Measured	Standard Deviation
R1 :	39	140	142	2.4
R2 :	40	70	69	1.3
R3 :	33	14.0	14.4	0.87
R4 :	33	7.0	7.2	0.59

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
23	0.0 - 4.0	0.37	18.51
20	4.0 - 10.0	0.40	5.77
11	10.0 - 20.0	0.44	2.91
8	20.0 - 100	1.8	2.95
2	100 - 200	0.7	0.47
64	Overall	0.8	N/A

DETECTION CRITERION: 0.6

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Long Term Blank :	27	0.06	0.050
Digested Blank :	34	0.65	0.305
Standard Cal :	27	403	47.5

PHOSPHORUS - FILTERED TOTAL

QUALITY CONTROL DATA FROM 07/01/83 TO 07/12/83

LAB: Rivers and Lakes

Analytical Range: 0.6 to 200 ug/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	30	150.0	150.8	0.8	1.17
B :	30	50.0	50.2	0.2	2.07
A+B :	30	200.0	201.0	1.0	2.47
A-B :	30	100.0	100.6	0.6	2.28
C :	28	15.0	15.2	0.2	0.32
D :	28	5.0	5.2	0.2	0.27
C+D :	28	20.0	20.4	0.4	0.37
C-D :	28	10.0	10.1	0.1	0.46

s.d(AB): Sw(within run): 1.61

S(between runs): 1.68

S/Sw: 1.04

s.d(CD): Sw(within run): 0.33

S(between runs): 0.30

S/Sw: 0.91

On any given day the calibration is accepted if the values obtained lie within the ranges:

191.0 to 209.0 for A+B

94.0 to 106.0 for A-B

18.7 to 21.4 for C+D

9.1 to 10.9 for C-D

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	49	140	141	1.3
R2 :	47	70	70	2.6
R3 :	53	14.0	13.1	3.04
R4 :	42	7.0	6.9	0.55

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
25	0.0 - 4.0	0.38	18.84
26	4.0 - 10.0	0.32	4.63
2	10.0 - 20.0	2.12	14.14
7	20.0 - 100	6.7	11.13
0	100 - 200	N/A	N/A
60	Overall	2.4	N/A

DETECTION CRITERION: 0.6

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	28	0.09	0.071
Digested Blank :	92	0.75	0.271
Standard Cal :	30	356	48.9

*** PHOSPHORUS - REACTIVE ORTHOPHOSPHATE ***

IDENTIFICATION:

Laboratory	: Rivers and Lakes	Method Introduced:	01/04/79
LIS Test Code Name:	PP04FR	Units	: mg/L as P
Work Station Code	: RNDNP	Unit Code	: 064815
Method Code	: 103DC2	Supervisor	: J. Crowther
Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.			

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Orthophosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance : 0.2 at the 0.13 mg/L as P level.

N.B. Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.
Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3	
Minimum Increment (W) : 0.0005	Detection Criterion (T): 0.003

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA
Drift : BL plus 1 standard

MODIFICATIONS:

01/02/84 - Sample filtration was eliminated for all sample classes but Great Lakes (G). Reduction period was reduced from 4 to 2 min. to lessen danger of poly phosphate conversion to orthophosphate during analysis.

15/05/84 - Microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration technique was changed from linear interpolation to the use of a quadratic.

01/10/84 - Sample filtration was eliminated for Great Lakes (G) samples.

PHOSPHORUS - REACTIVE ORTHOPHOSPHATE

QUALITY CONTROL DATA FROM 04/01/82 TO 30/12/82

LAB: Rivers and Lakes

Analytical Range: 0.002 to 0.100 mg/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
C :	204	0.0750	0.0748	-0.0002	0.00105
D :	204	0.0250	0.0246	-0.0004	0.00117
C+D :	203	0.1000	0.0994	-0.0006	0.00193
C-D :	203	0.0500	0.0501	0.0001	0.00107

s.d(CD): Sw(within run): 0.00076 S(between runs): 0.00111 S/Sw: 1.46

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.0955 to 0.1045 for C+D
0.0470 to 0.0530 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
233	0.000 - 0.020	0.0011	11.11
46	0.020 - 0.050	0.0025	7.02
21	0.050 - 0.100	0.0035	4.65
300	Overall	0.0017	N/A

DETECTION CRITERION: 0.002

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	204	0.0006	0.00053
Standard Cal :	203	754.9	28.49

PHOSPHORUS - REACTIVE ORTHOPHOSPHATE

QUALITY CONTROL DATA FROM 05/01/83 TO 31/01/84

LAB: Rivers and Lakes

Analytical Range: 0.002 to 0.100 mg/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
C :	195	0.0750	0.0746	-0.0004	0.00137
D :	195	0.0250	0.0251	0.0001	0.00065
C+D :	183	0.1000	0.0997	-0.0003	0.00170
C-D :	183	0.0500	0.0495	-0.0005	0.00129

s.d(CD): Sw(within run): 0.00092 S(between runs): 0.00107 S/Sw: 1.17

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.0955 to 0.1045 for C+D
0.0470 to 0.0530 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
362	0.000 - 0.010	0.0012	24.91
80	0.010 - 0.020	0.0020	13.30
78	0.020 - 0.050	0.0036	10.24
39	0.050 - 0.100	0.0019	2.57
559	Overall	0.0019	N/A

DETECTION CRITERION: 0.002

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	184	0.0003	0.00038
Standard Cal :	184	735	19.8

PHOSPHORUS - REACTIVE ORTHOPHOSPHATE
QUALITY CONTROL DATA FROM 23/08/84 TO 16/10/84

LAB: Rivers and Lakes

Analytical Range: 0.001 to 0.100 mg/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	32	0.1000	0.1002	0.0002	0.00122
B :	31	0.0250	0.0256	0.0006	0.00136
A+B :	31	0.1250	0.1258	0.0008	0.00208
A-B :	31	0.0750	0.0746	-0.0004	0.00156
C :	31	0.0250	0.0251	0.0001	0.00278
D :	31	0.0125	0.0129	0.0004	0.00103
C+D :	31	0.0375	0.0380	0.0005	0.00345
C-D :	31	0.0125	0.0123	-0.0002	0.00238

s.d(AB): Sw(within run): 0.00110

S(between runs): 0.00130

S/Sw: 1.17

s.d(CD): Sw(within run): 0.00168

S(between runs): 0.00210

S/Sw: 1.25

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.1205 to 0.1295 for A+B

0.0720 to 0.0780 for A-B

0.0330 to 0.0420 for C+D

0.0095 to 0.0155 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
55	0.000 - 0.010	0.0008	16.03
5	0.010 - 0.020	0.0008	5.48
8	0.020 - 0.050	0.0071	20.32
6	0.050 - 0.100	0.0016	2.10
74	Overall	0.0027	N/A

DETECTION CRITERION: 0.001

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	31	0.0010	0.00061

*** PHOSPHORUS - REACTIVE ORTHOPHOSPHATE ***

IDENTIFICATION:

Laboratory	: Sewage/Industrial	Method Introduced:	01/04/79
LIS Test Name Code:	PP04FR	Units	: mg/L as P
Work Station Code	: SNH3P	Unit Code	: 064815
Method Code	: 103BC2	Supervisor	: P. Campbell
Sample Type/Matrix:	Sewage, Industrial Waste, Leachate, Domestic Waters, Effluents		

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Orthophosphate is determined on an automatically filtered sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance : 0.4 at the 4.0 mg/L as P level.

N.B. Ammonia plus ammonium is determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W) : 0.01

Detection Criterion (T): 0.06

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : LTBL plus 4 standards, eg, QCA

Drift : BL plus 1 standard

PHOSPHORUS - REACTIVE ORTHOPHOSPHATE

QUALITY CONTROL DATA FROM 07/01/82 TO 22/12/82

LAB: Sewage/Industrial

Analytical Range: 0.02 to 10.00 mg/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	128	7.00	7.05	0.05	0.092
B :	128	3.50	3.54	0.04	0.061
A+B :	127	10.50	10.59	0.09	0.136
A-B :	127	3.50	3.51	0.01	0.075
C :	128	3.50	3.52	0.02	0.057
D :	128	0.70	0.70	-0.00	0.022
C+D :	127	4.20	4.21	0.01	0.070
C-D :	127	2.80	2.82	0.02	0.051

s.d(AB): Sw(within run): 0.053

S(between runs): 0.078

S/Sw: 1.47

s.d(CD): Sw(within run): 0.036

S(between runs): 0.043

S/Sw: 1.20

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.05	to	10.95	for A+B
3.20	to	3.80	for A-B
4.02	to	4.38	for C+D
2.68	to	2.92	for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
95	0.00 - 0.80	0.013	3.36
23	0.80 - 2.00	0.042	2.98
34	2.00 - 4.00	0.037	1.23
30	4.00 - 7.00	0.186	3.39
1	7.00 - 10.00	N/A	N/A
183	Overall	0.086	N/A

DETECTION CRITERION: 0.02

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	128	4.93	1.551

PHOSPHORUS - REACTIVE ORTHOPHOSPHATE

QUALITY CONTROL DATA FROM 05/01/83 TO 13/12/83

LAB: Sewage/Industrial

Analytical Range: 0.06 to 10.00 mg/L as P

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	106	7.00	7.01	0.01	0.077
B :	106	3.50	3.55	0.05	0.048
A+B :	105	10.50	10.56	0.06	0.110
A-B :	105	3.50	3.46	-0.04	0.067
C :	106	3.50	3.51	0.01	0.041
D :	104	0.70	0.69	-0.01	0.015
C+D :	103	4.20	4.20	0.00	0.047
C-D :	103	2.80	2.82	0.02	0.042

s.d(AB): Sw(within run): 0.047

S(between runs): 0.064

S/Sw: 1.36

s.d(CD): Sw(within run): 0.030

S(between runs): 0.031

S/Sw: 1.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.05	to	10.95	for A+B
3.20	to	3.80	for A-B
4.02	to	4.38	for C+D
2.68	to	2.92	for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
91	0.00 - 0.80	0.035	8.86
51	0.80 - 2.00	0.033	2.34
63	2.00 - 4.00	0.058	1.94
23	4.00 - 7.00	0.073	1.33
1	7.00 - 10.00	N/A	N/A
229	Overall	0.050	N/A

DETECTION CRITERION: 0.06

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Standard Cal :	106	4.02	0.421

PHOSPHORUS - REACTIVE ORTHOPHOSPHATE

QUALITY CONTROL DATA FROM 11/01/84 TO 19/12/84

LAB: Sewage/Industrial

Analytical Range: 0.06 to 10.00 mg/L as P

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	107	7.00	7.05	0.05	0.067
B :	107	3.50	3.55	0.05	0.055
A+B :	106	10.50	10.61	0.11	0.108
A-B :	106	3.50	3.50	0.00	0.058
C :	111	3.50	3.52	0.02	0.042
D :	109	0.70	0.68	-0.02	0.034
C+D :	108	4.20	4.20	0.00	0.062
C-D :	108	2.80	2.84	0.04	0.046

s.d(AB): Sw(within run): 0.041

S(between runs): 0.061

S/Sw: 1.49

s.d(CD): Sw(within run): 0.033

S(between runs): 0.038

S/Sw: 1.17

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.05	to	10.95	for A+B
3.20	to	3.80	for A-B
4.02	to	4.38	for C+D
2.68	to	2.92	for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
146	0.00 - 0.80	0.039	9.69
19	0.80 - 2.00	0.183	13.04
15	2.00 - 4.00	0.090	2.99
11	4.00 - 7.00	0.128	2.32
2	7.00 - 10.00	0.071	0.83
193	Overall	0.088	N/A

DETECTION CRITERION: 0.06

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Standard Cal :	94	4.86	0.915

*** PHOSPHORUS - TOTAL ***

Laboratory : Dorset Method Introduced: 22/03/79
Supervisor : F. Tomassini Units : ug/L as P
Sample Type/Matrix: Streams, Lakes, Precipitation

SAMPLING:

Quantity Required: 35 mL
Container : Specially marked Pyrex culture tubes with Teflon-lined caps

ANALYTICAL PROCEDURE:

After withdrawal of excess volume, digestion reagent is added and samples are autoclaved in sulphuric acid-potassium persulphate media at 121 C for 60 min. The orthophosphate content of the digestate is determined colourimetrically by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.
Approximate absorbance : 0.3 at the 200 ug/L as P level.

INSTRUMENTATION:

Autoclave plus basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube. Two analytical ranges are obtained from the output of the colourimeter.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.1 Detection Criterion (T): 0.2

CALIBRATION:

BL plus 2 undigested standards

CONTROLS:

Calibration : LTBL plus 4 undigested standards, eg, QCA
Recovery : 3 digested BL plus 4 digested standards, eg, R1
Drift : BL plus 2 undigested standards

NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

PHOSPHORUS - TOTAL

QUALITY CONTROL DATA FROM 13/01/82 TO 20/12/82

LAB: Dorset

Analytical Range: 0.2 to 200 ug/L as P

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	172	171	170	-1	1.0
B :	172	57	58	1	0.8
A+B :	171	228	228	-0	1.1
A-B :	171	114	112	-2	1.4
C :	172	17.1	17.5	0.4	0.41
D :	172	5.7	5.8	0.1	0.22
C+D :	171	22.8	23.3	0.5	0.57
C-D :	171	11.4	11.6	0.2	0.33

s.d(AB): Sw(within run): 1.0 S(between runs): 0.9 S/Sw: 0.90
s.d(CD): Sw(within run): 0.23 S(between runs): 0.33 S/Sw: 1.40

On any given day the calibration is accepted if the values obtained lie within the ranges:

219 to 237 for A+B
108 to 120 for A-B
21.9 to 23.7 for C+D
10.8 to 12.0 for C-D

RECOVERIES:

	Number of Data	Expected Concn	Av. Concn Measured	Standard Deviation
R1 :	343	140	139	3.8
R2 :	344	70	70	1.5
R3 :	338	14.0	13.9	0.70
R4 :	343	7.0	7.0	0.89

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
12	0.0 - 2.0	0.12	11.65
285	2.0 - 10.0	0.26	4.30
132	10.0 - 20.0	0.40	2.70
76	20.0 - 100	1.0	1.69
7	100 - 200	1.1	0.71
512	Overall	0.5	N/A

DETECTION CRITERION: 0.2

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Long Term Blank :	132	0.1	0.11
Digested Blank :	173	1.1	0.42
Standard Cal :	171	5.53	0.518

PHOSPHORUS - TOTAL

QUALITY CONTROL DATA FROM 06/01/83 TO 22/12/83

LAB: Dorset

Analytical Range: 0.2 to 200 ug/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	180	171	171	-0	0.9
B :	180	57	57	0	0.5
A+B :	179	228	228	0	1.2
A-B :	179	114	113	-1	0.8
C :	180	17.1	17.2	0.1	0.11
D :	180	5.7	5.7	-0.0	0.08
C+D :	179	22.8	22.9	0.1	0.15
C-D :	179	11.4	11.5	0.1	0.11

s.d(AB): Sw(within run): 0.6 S(between runs): 0.7 S/Sw: 1.22
s.d(CD): Sw(within run): 0.08 S(between runs): 0.09 S/Sw: 1.21

On any given day the calibration is accepted if the values obtained lie within the ranges:

219 to 237 for A+B
108 to 120 for A-B
21.9 to 23.7 for C+D
10.8 to 12.0 for C-D

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	172	140	140	1.2
R2 :	172	70	70	0.7
R3 :	172	14.0	14.2	0.47
R4 :	171	7.0	7.0	0.17

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
24	0.0 - 2.0	0.10	10.49
283	2.0 - 10.0	0.13	2.23
157	10.0 - 20.0	0.30	1.99
68	20.0 - 100	0.8	1.38
5	100 - 200	0.9	0.60
537	Overall	0.4	N/A

DETECTION CRITERION: 0.2

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	180	0.1	0.06
Digested Blank :	179	0.6	0.18
Standard Cal :	180	6.17	0.136

PHOSPHORUS - TOTAL

QUALITY CONTROL DATA FROM 04/01/84 TO 20/12/84

LAB: Dorset

Analytical Range: 0.5 to 200 ug/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	181	171	172	1	0.9
B :	181	57	57	0	0.5
A+B :	180	228	229	1	1.1
A-B :	180	114	115	1	0.9
C :	181	17.1	17.1	-0.0	0.96
D :	181	5.7	5.7	-0.0	0.16
C+D :	180	22.8	22.8	0.0	0.86
C-D :	180	11.4	11.4	0.0	0.87

s.d(AB): Sw(within run): 0.6 S(between runs): 0.7 S/Sw: 1.09
s.d(CD): Sw(within run): 0.62 S(between runs): 0.69 S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

219 to 237 for A+B
108 to 120 for A-B
21.9 to 23.7 for C+D
10.8 to 12.0 for C-D

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	181	140	141	1.8
R2 :	181	70	70	0.8
R3 :	181	14.0	14.1	0.28
R4 :	180	7.0	7.0	0.24

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
6	0.0 - 2.0	0.31	31.25
244	2.0 - 10.0	3.84	64.00
152	10.0 - 20.0	0.33	2.21
127	20.0 - 100	0.7	1.12
9	100 - 200	0.9	0.59
538	Overall	2.6	N/A

DETECTION CRITERION: 0.5

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	178	0.0	0.03
Digested Blank :	181	0.7	0.40
Standard Cal :	181	5.84	0.848

*** PHOSPHORUS-TOTAL ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/79
LIS Test Name Code: PPUT Units : mg/L as P
Work Station Code : RTNP Unit Code : 064815
Method Code : 504AC2 Supervisor : J. Crowther
Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters kept at 200 C and 360 C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance : 0.4 at the 0.20 mg/L as P level.

N.B. Total Kjeldahl nitrogen is determined simultaneously.

INSTRUMENTATION:

- Block digesters(2)
- Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.
- Data capture, reduction, and processing via a multi-stage microcomputer system

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W) : 0.001

Detection Criterion (T): 0.005

CALIBRATION:

BL plus 4 undigested standards

CONTROLS:

Calibration : LTBL plus 2 undigested standards, eg, QCA

Recovery : 3 digested BL plus 3 digested standards in duplicate, eg, R1

Drift : BL plus 1 undigested standard

MODIFICATIONS:

15/08/83 - Microcomputer system was introduced. At this time the calibration technique was changed from linear interpolation to the use of a quadratic.

NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

PHOSPHORUS - TOTAL

QUALITY CONTROL DATA FROM 05/01/82 TO 23/12/82

LAB: Rivers and Lakes

Analytical Range: 0.005 to 0.200 mg/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	158	0.1500	0.1504	0.0004	0.00098
B :	158	0.0500	0.0503	0.0003	0.00071
A+B :	157	0.2000	0.2006	0.0006	0.00147
A-B :	157	0.1000	0.1001	0.0001	0.00087

s.d(AB): Sw(within run): 0.00062 S(between runs): 0.00086 S/Sw: 1.39

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.1910 to 0.2090 for A+B
0.0940 to 0.1060 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	301	0.140	0.141	0.0051
R2 :	299	0.080	0.085	0.0040
R3 :	290	0.030	0.030	0.0035

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
420	0.000 - 0.040	0.0030	14.98
67	0.040 - 0.100	0.0027	3.83
24	0.100 - 0.200	0.0040	2.70
511	Overall	0.0030	N/A

DETECTION CRITERION: 0.005

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	157	0.0005	0.00050
Digested Blank :	276	0.0035	0.00331
Standard Cal :	158	410.1	12.68

PHOSPHORUS - TOTAL

QUALITY CONTROL DATA FROM 05/01/83 TO 05/12/83

LAB: Rivers and Lakes

Analytical Range: 0.006 to 0.200 mg/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	141	0.1500	0.1490	-0.0010	0.00149
B :	141	0.0500	0.0496	-0.0004	0.00105
A+B :	140	0.2000	0.1987	-0.0014	0.00223
A-B :	140	0.1000	0.0994	-0.0006	0.00129

s.d(AB): Sw(within run): 0.00091 S(between runs): 0.00129 S/Sw: 1.42

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.1910 to 0.2090 for A+B
0.0940 to 0.1060 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	269	0.140	0.135	0.0050
R2 :	270	0.080	0.082	0.0041
R3 :	269	0.030	0.028	0.0024

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
233	0.000 - 0.040	0.0034	16.89
48	0.040 - 0.100	0.0052	7.37
15	0.100 - 0.200	0.0064	4.29
296	Overall	0.0040	N/A

DETECTION CRITERION: 0.006

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	139	0.0006	0.00097
Digested Blank :	349	0.0026	0.00179
Standard Cal :	66	400	17.4

PHOSPHORUS - TOTAL

QUALITY CONTROL DATA FROM 03/01/84 TO 31/12/84

LAB: Rivers and Lakes

Analytical Range: 0.004 to 0.200 mg/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	156	0.1500	0.1484	-0.0016	0.00106
B :	156	0.0500	0.0494	-0.0006	0.00099
A+B :	155	0.2000	0.1977	-0.0023	0.00155
A-B :	155	0.1000	0.0990	-0.0010	0.00135

s.d(AB): Sw(within run): 0.00095 S(between runs): 0.00102 S/Sw: 1.07

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.1910 to 0.2090 for A+B
0.0940 to 0.1060 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	307	0.140	0.136	0.0175
R2 :	305	0.080	0.082	0.0054
R3 :	303	0.030	0.029	0.0035

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
256	0.000 - 0.040	0.0022	11.07
52	0.040 - 0.100	0.0041	5.85
18	0.100 - 0.200	0.0053	3.52
326	Overall	0.0029	N/A

DETECTION CRITERION: 0.004

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	156	0.0010	0.00016
Digested Blank :	156	0.0026	0.00181
Standard Cal :	58	388	16.1

*** PHOSPHORUS - TOTAL ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: 01/04/79
LIS Test Name Code: PPUT Units : mg/L as P
Work Station Code : STKNP Unit Code : 064815
Method Code : 504BC2 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Domestic Waters, Effluents,
Leachates

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters kept at 200 C and 360 C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance : 0.4 at the 2.0 mg/L as P level.

N.B. Total Kjeldahl nitrogen is determined simultaneously.

INSTRUMENTATION:

- Block digesters(2)
- Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

REPORTING:

Maximum Significant Figures: 3

Minimum Increment (W) : 0.005

Detection Criterion (T): 0.07

CALIBRATION:

BL plus 1 undigested standard

CONTROLS:

Calibration : LTBL plus 2 undigested standards, eg, QCA

Recovery : 2 digested BL plus 2 digested standards in duplicate, eg, R1

Drift : BL plus 1 undigested standard

NOTES:

System is calibrated with undigested standards.

PHOSPHORUS - TOTAL

QUALITY CONTROL DATA FROM 05/01/82 TO 29/12/82

LAB: Sewage/Industrial

Analytical Range: 0.01 to 2.00 mg/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	135	1.40	1.40	-0.00	0.014
B :	113	0.28	0.28	-0.00	0.009
A+B :	106	1.68	1.67	-0.01	0.016
A-B :	106	1.12	1.12	0.00	0.018

s.d(AB): Sw(within run): 0.013 S(between runs): 0.012 S/Sw: 0.90

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.59 to 1.77 for A+B
1.06 to 1.18 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	107	1.40	1.40	0.016
R2 :	100	0.70	0.70	0.010

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
36	0.00 - 0.40	0.009	4.29
64	0.40 - 1.00	0.010	1.41
38	1.00 - 2.00	0.016	1.09
138	Overall	0.012	N/A

DETECTION CRITERION: 0.01

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	152	4.28	0.287

PHOSPHORUS - TOTAL

QUALITY CONTROL DATA FROM 07/01/83 TO 22/12/83

LAB: Sewage/Industrial

Analytical Range: 0.02 to 2.00 mg/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	116	1.40	1.42	0.02	0.029
B :	115	0.28	0.28	-0.00	0.013
A+B :	113	1.68	1.69	0.01	0.036
A-B :	113	1.12	1.14	0.02	0.025

s.d(AB): Sw(within run): 0.018

S(between runs): 0.022

S/Sw: 1.24

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.59 to 1.77 for A+B
1.06 to 1.18 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	110	1.40	1.41	0.037
R2 :	110	0.70	0.70	0.030

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
37	0.00 - 0.40	0.015	7.59
53	0.40 - 1.00	0.027	3.86
35	1.00 - 2.00	0.046	3.08
125	Overall	0.033	N/A

DETECTION CRITERION: 0.02

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Standard Cal :	119	4.38	0.314

PHOSPHORUS - TOTAL

QUALITY CONTROL DATA FROM 05/01/84 TO 28/12/84

LAB: Sewage/Industrial

Analytical Range: 0.04 to 2.00 mg/L as P

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	139	1.40	1.40	0.00	0.028
B :	137	0.28	0.27	-0.01	0.019
A+B :	136	1.68	1.67	-0.01	0.036
A-B :	136	1.12	1.14	0.02	0.032

s.d(AB): Sw(within run): 0.023 S(between runs): 0.024 S/Sw: 1.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.59 to 1.77 for A+B
1.06 to 1.18 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	121	1.40	1.39	0.023
R2 :	121	0.70	0.68	0.025

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
199	0.00 - 0.40	0.022	10.88
52	0.40 - 1.00	0.064	9.08
16	1.00 - 2.00	0.121	8.06
267	Overall	0.048	N/A

DETECTION CRITERION: 0.04

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Digested Blank :	87	0.008	0.0058
Standard Cal :	128	4.51	0.313

*** POTASSIUM ***

IDENTIFICATION:

Laboratory	: Domestic Water	Method Introduced:	Before '74
LIS Test Name Code:	KKUR	Units	: mg/L as K
Work Station Code	: WNAK	Unit Code	: 064819
Method Code	: 002BA1	Supervisor	: M. Rawlings
Sample Type/Matrix: Domestic Waters, Leachates, Effluents			

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm using an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train.
Approximate absorbance: 0.15 at 10 mg/L as K level.

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS). Two analytical ranges are obtained from the output of the AAS.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.05 Detection Criterion (T):0.1

CALIBRATION:

BL plus 2 standards.

CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA
Drift : BL plus 3 standards

POTASSIUM

QUALITY CONTROL DATA FROM 16/03/82 TO 30/12/82

LAB: Domestic Water

Analytical Range: 0.11 to 40.0 mg/L as K

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	70	26.0	25.8	-0.2	0.62
B :	69	6.5	6.5	-0.0	0.23
A+B :	69	32.5	32.3	-0.2	0.68
A-B :	69	19.5	19.4	-0.1	0.63
C :	70	6.50	6.47	-0.03	0.154
D :	70	1.30	1.30	0.00	0.092
C+D :	70	7.80	7.78	-0.02	0.182
C-D :	70	5.20	5.17	-0.03	0.176

s.d(AB): Sw(within run): 0.45 S(between runs): 0.47 S/Sw: 1.04
s.d(CD): Sw(within run): 0.124 S(between runs): 0.127 S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

30.7 to 34.3 for A+B
18.3 to 20.7 for A-B
7.35 to 8.25 for C+D
4.90 to 5.50 for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
87	0.00 - 2.00	0.068	6.81
42	2.00 - 5.00	0.095	2.71
19	5.00 - 10.00	0.185	2.47
9	10.00 - 20.0	0.13	0.87
6	20.0 - 40.0	0.60	1.99
163	Overall	0.22	N/A

DETECTION CRITERION: 0.11

POTASSIUM

QUALITY CONTROL DATA FROM 13/01/83 TO 21/12/83

LAB: Domestic Water

Analytical Range: 0.11 to 40.0 mg/L as K

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	76	26.0	25.9	-0.1	0.39
B :	76	6.5	6.4	-0.1	0.22
A+B :	76	32.5	32.4	-0.1	0.42
A-B :	76	19.5	19.5	-0.0	0.47
C :	76	6.50	6.48	-0.02	0.152
D :	75	1.30	1.27	-0.03	0.080
C+D :	75	7.80	7.75	-0.05	0.196
C-D :	75	5.20	5.21	0.01	0.145

s.d(AB): Sw(within run): 0.33 S(between runs): 0.32 S/Sw: 0.94
s.d(CD): Sw(within run): 0.103 S(between runs): 0.122 S/Sw: 1.19

On any given day the calibration is accepted if the values obtained lie within the ranges:

30.7 to 34.3 for A+B
18.3 to 20.7 for A-B
7.35 to 8.25 for C+D
4.90 to 5.50 for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
131	0.00 - 2.00	0.067	6.73
47	2.00 - 5.00	0.153	4.38
11	5.00 - 10.00	0.238	3.17
5	10.00 - 20.0	0.30	1.98
4	20.0 - 40.0	0.76	2.54
198	Overall	0.21	N/A

DETECTION CRITERION: 0.11

POTASSIUM

QUALITY CONTROL DATA FROM 03/01/84 TO 11/12/84

LAB: Domestic Water

Analytical Range: 0.11 to 40.0 mg/L as K

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	95	26.0	25.9	-0.1	0.30
B :	95	6.5	6.5	-0.0	0.18
A+B :	95	32.5	32.4	-0.1	0.35
A-B :	95	19.5	19.5	-0.0	0.35
C :	95	6.50	6.50	0.00	0.104
D :	95	1.30	1.32	0.02	0.071
C+D :	95	7.80	7.82	0.02	0.122
C-D :	95	5.20	5.19	-0.01	0.131

s.d(AB): Sw(within run): 0.25

S(between runs): 0.25

S/Sw: 0.99

s.d(CD): Sw(within run): 0.093

S(between runs): 0.089

S/Sw: 0.96

On any given day the calibration is accepted if the values obtained lie within the ranges:

30.7 to 34.3 for A+B
18.3 to 20.7 for A-B
7.35 to 8.25 for C+D
4.90 to 5.50 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
160	0.00 - 2.00	0.068	6.77
75	2.00 - 5.00	0.118	3.38
23	5.00 - 10.00	0.121	1.62
8	10.00 - 20.0	0.31	2.08
4	20.0 - 40.0	0.18	0.61
270	Overall	0.12	N/A

DETECTION CRITERION: 0.11

*** POTASSIUM ***

IDENTIFICATION:

Laboratory	: Precipitation	Method Introduced:	18/05/79
LIS Test Name Code:	KKUR	Units	: mg/L as K
Work Station Code	: PRAA	Unit Code	: 064819
Method Code	: 002EA1	Supervisor	: M. Rawlings
Sample Type/Matrix: Precipitation, Throughfall, Stemflow.			

SAMPLING:

Quantity Required: 25 mL
Container : Polystyrene

ANALYTICAL PROCEDURE:

Samples are analysed by AAS at 766.5 nm with an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train.
Approximate absorbance: 0.5 at the 1.00 mg/L level.

INSTRUMENTATION:

Automated modular continuous flow atomic absorption spectrophotometer (AAS) system

REPORTING:

Maximum Significant Figures: 3	
Minimum Increment (W) : 0.005	Detection Criterion (T): 0.014

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : BL plus 2 standards every 10 samples

POTASSIUM

QUALITY CONTROL DATA FROM 01/01/82 TO 23/12/82

LAB: Precipitation

Analytical Range: 0.01 to 1.00 mg/L as K

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	89	0.60	0.60	-0.00	0.009
B :	89	0.10	0.09	-0.01	0.010
A+B :	89	0.70	0.69	-0.01	0.014
A-B :	89	0.50	0.51	0.01	0.012

s.d(AB): Sw(within run): 0.009 S(between runs): 0.009 S/Sw: 1.10

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.66 to 0.75 for A+B
0.47 to 0.53 for A-B

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
70	0.00 - 0.20	0.008	8.50
11	0.20 - 0.50	0.014	3.90
5	0.50 - 1.00	0.044	5.86
86	Overall	0.014	N/A

DETECTION CRITERION: 0.01

POTASSIUM

QUALITY CONTROL DATA FROM 06/01/83 TO 29/12/83

LAB: Precipitation

Analytical Range: 0.01 to 1.00 mg/L as K

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	46	0.60	0.61	0.01	0.009
B :	46	0.10	0.10	-0.00	0.007
A+B :	46	0.70	0.70	0.00	0.012
A-B :	46	0.50	0.51	0.01	0.011

s.d(AB): Sw(within run): 0.008

S(between runs): 0.008

S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.66 to 0.75 for A+B
0.47 to 0.53 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
8	0.00 - 0.20	0.006	5.63
0	0.20 - 0.50	N/A	N/A
0	0.50 - 1.00	N/A	N/A
8	Overall	0.006	N/A

DETECTION CRITERION: 0.01

POTASSIUM

QUALITY CONTROL DATA FROM 04/01/84 TO 19/12/84

LAB: Precipitation

Analytical Range: 0.01 to 1.00 mg/L as K

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	51	0.60	0.61	0.01	0.009
B :	51	0.10	0.09	-0.01	0.006
A+B :	51	0.70	0.70	0.00	0.012
A-B :	51	0.50	0.52	0.02	0.010

s.d(AB): Sw(within run): 0.007 S(between runs): 0.008 S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.66 to 0.75 for A+B
0.47 to 0.53 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
76	0.00 - 0.20	0.007	6.74
3	0.20 - 0.50	0.013	3.60
2	0.50 - 1.00	0.007	0.94
81	Overall	0.007	N/A

DETECTION CRITERION: 0.01

*** POTASSIUM ***

IDENTIFICATION:

Laboratory	: Rivers and Lakes	Method Introduced:	01/04/74
LIS Test Name Code:	KKUR	Units	: mg/L as K
Work Station Code	: RMNAKH,RMNAKL	Unit Code	: 064819
Method Code	: 002CA1,002DA1	Supervisor	: J. Crowther
Sample Type/Matrix:	Rivers, Lakes, Soil Extracts, Effluents.		

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm using an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train.
Approximate absorbance: RMNAKH: 0.9, RMNAKL: 0.5; full scale values

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS).

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.02, 0.01 Detection Criterion (T):0.04, 0.03

CALIBRATION:

BL plus 10 standards

CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA for each analytical range
Drift : BL plus 1 standard for each analytical range

MODIFICATIONS:

01/12/81- Calibration range became 5.00 mg/L full scale; second analytical range was dropped.
01/03/84- Analytical range(RMNAKL) was added; full scale:1.00 mg/L. This range is currently restricted to special programs.
01/09/84- Analytical range(RMNAKH) was increased from 5.00 to 10.0 mg/L full scale. Calibration technique was changed from quadratric to linear interpolation. Sodium is no longer determined simultaneously.

POTASSIUM

QUALITY CONTROL DATA FROM 15/01/82 TO 22/12/82

LAB: Rivers and Lakes

Analytical Range: 0.04 to 5.00 mg/L as K

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	48	3.50	3.45	-0.05	0.062
B :	48	1.40	1.34	-0.06	0.046
A+B :	48	4.90	4.80	-0.10	0.094
A-B :	48	2.10	2.11	0.01	0.054

s.d(AB): Sw(within run): 0.038 S(between runs): 0.055 S/Sw: 1.43

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.68 to 5.13 for A+B
1.95 to 2.25 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
119	0.00 - 1.00	0.023	4.60
33	1.00 - 2.50	0.038	2.17
9	2.50 - 5.00	0.152	4.05
161	Overall	0.052	N/A

DETECTION CRITERION: 0.04

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	25	0.00	0.000
Full Scale Abs :	47	0.956	0.2120

POTASSIUM

QUALITY CONTROL DATA FROM 07/01/83 TO 22/12/83

LAB: Rivers and Lakes

Analytical Range: 0.03 to 5.00 mg/L as K

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	67	3.50	3.49	-0.01	0.047
B :	67	1.40	1.38	-0.02	0.031
A+B :	67	4.90	4.87	-0.03	0.064
A-B :	67	2.10	2.11	0.01	0.048

s.d(AB): Sw(within run): 0.034 S(between runs): 0.040 S/Sw: 1.18

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.68 to 5.13 for A+B
1.95 to 2.25 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
181	0.00 - 1.00	0.017	3.48
27	1.00 - 2.50	0.025	1.43
16	2.50 - 5.00	0.076	2.02
224	Overall	0.030	N/A

DETECTION CRITERION: 0.03

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	67	0.00	0.000
Full Scale Abs :	62	0.629	0.1414

POTASSIUM

QUALITY CONTROL DATA FROM 05/01/84 TO 28/08/84

LAB: Rivers and Lakes

Analytical Range: 0.03 to 5.00 mg/L as K

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	41	3.50	3.47	-0.03	0.071
B :	40	1.40	1.40	-0.00	0.031
A+B :	40	4.90	4.87	-0.03	0.088
A-B :	40	2.10	2.08	-0.02	0.067

s.d(AB): Sw(within run): 0.047

S(between runs): 0.055

S/Sw: 1.16

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.68 to 5.13 for A+B
1.95 to 2.25 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
90	0.00 - 1.00	0.021	4.21
29	1.00 - 2.50	0.031	1.80
16	2.50 - 5.00	0.059	1.57
135	Overall	0.032	N/A

DETECTION CRITERION: 0.03

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	40	0.00	0.004
Full Scale Abs :	41	0.535	0.0962

POTASSIUM

QUALITY CONTROL DATA FROM 27/02/84 TO 30/08/84

LAB: Rivers and Lakes

Analytical Range: 0.04 to 1.00 mg/L as K

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	33	0.70	0.70	0.00	0.023
B :	32	0.28	0.29	0.01	0.017
A+B :	32	0.98	0.99	0.01	0.038
A-B :	32	0.42	0.42	-0.00	0.013

s.d(AB): Sw(within run): 0.010 S(between runs): 0.020 S/Sw: 2.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.94 to 1.03 for A+B
0.39 to 0.45 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
0	0.00 - 0.10	N/A	N/A
55	0.10 - 0.40	0.024	9.66
61	0.40 - 1.00	0.015	2.13
116	Overall	0.020	N/A

DETECTION CRITERION: 0.04

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	32	0.00	0.000
High Std ABS :	33	0.368	0.0426

*** SILICON - REACTIVE SILICATES ***

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/02/75
LIS Test Name Code: SI03UR Units : mg/L as Si
Work Station Code : RMSICL Unit Code : 064814
Method Code : 001BC2 Supervisor : J. Crowther
Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,
Domestic Water Supplies, Leachates

SAMPLING:

Quantity Required: 50 mL
Container : Plastic

ANALYTICAL PROCEDURE:

Reactive silicates are determined by formation of a reduced molybdo-silicate complex at pH 1.6, using ascorbic acid as the reducing agent, and oxalic acid to suppress phosphate interference.
Approximate absorbance: 0.7 at the 5.0 mg/L as Si level.
N.B. Chloride is determined simultaneously.

INSTRUMENTATION:

Boxed-FIA system consisting of basic automated modular continuous flow system plus the following modules: sample injection valve with air-flow controls, timer, bubble-gate. Colourimetric measurement is through a 5.0 cm. light path at 660 nm.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.02 Detection Criterion (T): 0.06

CALIBRATION:

BL plus 10 standards

CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA
Drift : BL plus 3 standard

MODIFICATIONS:

04/07/83 - Modules required for Boxed-FIA system were introduced. The number of calibration standards was increased from 2 to 10. The analytical rate was tripled.

NOTES:

Calibration standard is a hydrate: $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$

SILICON - REACTIVE SILICATES

QUALITY CONTROL DATA FROM 04/01/82 TO 22/12/82

LAB: Rivers and Lakes

Analytical Range: 0.07 to 5.00 mg/L as Si

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	130	2.50	2.57	0.07	0.064
B :	130	0.50	0.50	0.00	0.019
A+B :	129	3.00	3.07	0.07	0.071
A-B :	129	2.00	2.07	0.07	0.062

s.d(AB): Sw(within run): 0.044 S(between runs): 0.047 S/Sw: 1.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.78 to 3.23 for A+B
1.85 to 2.15 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
211	0.00 - 1.00	0.041	8.19
137	1.00 - 2.50	0.063	3.58
61	2.50 - 5.00	0.118	3.14
409	Overall	0.068	N/A

DETECTION CRITERION: 0.07

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	115	0.01	0.015
Standard Cal :	130	180.2	17.48

SILICON - REACTIVE SILICATES

QUALITY CONTROL DATA FROM 06/01/83 TO 30/09/83

LAB: Rivers and Lakes

Analytical Range: 0.06 to 5.00 mg/L as Si

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	78	2.50	2.48	-0.02	0.051
B :	78	0.50	0.50	-0.00	0.028
A+B :	78	3.00	2.98	-0.02	0.055
A-B :	78	2.00	1.98	-0.02	0.062

s.d(AB): Sw(within run): 0.044 S(between runs): 0.041 S/Sw: 0.95

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.78 to 3.23 for A+B
1.85 to 2.15 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
103	0.00 - 1.00	0.034	6.80
87	1.00 - 2.50	0.046	2.64
33	2.50 - 5.00	0.042	1.12
223	Overall	0.041	N/A

DETECTION CRITERION: 0.06

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	76	0.00	0.013
Standard Cal :	77	193	21.6

SILICON - REACTIVE SILICATES

QUALITY CONTROL DATA FROM 07/10/83 TO 19/12/84

LAB: Rivers and Lakes

Analytical Range: 0.05 to 5.00 mg/L as Si

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	98	3.00	3.04	0.04	0.084
B :	98	0.80	0.83	0.03	0.050
A+B :	97	3.80	3.87	0.07	0.111
A-B :	97	2.20	2.21	0.01	0.081

s.d(AB): Sw(within run): 0.058

S(between runs): 0.069

S/Sw: 1.20

On any given day the calibration is accepted if the values obtained lie within the ranges:

3.58 to 4.03 for A+B
2.05 to 2.35 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
177	0.00 - 1.00	0.032	6.47
87	1.00 - 2.50	0.117	6.68
55	2.50 - 5.00	0.126	3.35
319	Overall	0.091	N/A

DETECTION CRITERION: 0.05

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	98	0.01	0.006
Standard Cal :	96	321	49.0

*** SODIUM ***

IDENTIFICATION:

Laboratory	: Domestic Water	Method Introduced:	Before '74
LIS Test Name Code:	NAUR	Units	: mg/L as Na
Work Station Code	: WNAK	Unit Code	: 064811
Method Code	: 002BA1	Supervisor	: M. Rawlings
Sample Type/Matrix: Domestic Waters, Leachates, Effluents			

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm using an air-acetylene flame. Potassium is added as a suppressant via an automated sampling train.
Approximate absorbance: 0.16 at the 50 mg/L level.

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS). Two analytical ranges are obtained from the output of the AAS.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.1 Detection Criterion (T):0.6

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA
Drift : BL plus 3 standards

SODIUM

QUALITY CONTROL DATA FROM 06/01/82 TO 20/12/82

LAB: Domestic Water

Analytical Range: 0.8 to 200 mg/L as Na

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	91	130	130	0	2.4
B :	91	33	33	0	1.0
A+B :	91	163	163	0	2.8
A-B :	91	98	97	-0	2.5
C :	90	32.5	33.0	0.5	0.89
D :	90	6.5	6.6	0.1	0.33
C+D :	90	39.0	39.6	0.6	1.09
C-D :	90	26.0	26.4	0.4	0.77

s.d(AB): Sw(within run): 1.8

S(between runs): 1.9

S/Sw: 1.05

s.d(CD): Sw(within run): 0.54

S(between runs): 0.67

S/Sw: 1.23

On any given day the calibration is accepted if the values obtained lie within the ranges:

155 to 170 for A+B
93 to 103 for A-B
36.8 to 41.3 for C+D
24.5 to 27.5 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
97	0.0 - 10.0	0.48	9.63
51	10.0 - 25.0	0.81	4.66
25	25.0 - 50.0	1.30	3.48
16	50.0 - 100	1.1	1.52
8	100 - 200	5.1	3.43
197	Overall	1.5	N/A

DETECTION CRITERION: 0.8

SODIUM

QUALITY CONTROL DATA FROM 13/01/83 TO 21/12/83

LAB: Domestic Water

Analytical Range: 0.6 to 200 mg/L as Na

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	73	130	131	1	2.1
B :	73	33	33	0	1.1
A+B :	73	163	164	1	2.1
A-B :	73	98	98	1	2.6
C :	73	32.5	33.2	0.7	0.65
D :	73	6.5	6.5	0.0	0.26
C+D :	73	39.0	39.7	0.7	0.75
C-D :	73	26.0	26.6	0.6	0.66

s.d(AB): Sw(within run): 1.9

S(between runs): 1.7

S/Sw: 0.91

s.d(CD): Sw(within run): 0.47

S(between runs): 0.50

S/Sw: 1.07

On any given day the calibration is accepted if the values obtained lie within the ranges:

155 to 170 for A+B

93 to 103 for A-B

36.8 to 41.3 for C+D

24.5 to 27.5 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
80	0.0 - 10.0	0.37	7.32
46	10.0 - 25.0	0.32	1.82
23	25.0 - 50.0	1.54	4.09
27	50.0 - 100	1.9	2.58
16	100 - 200	1.9	1.28
192	Overall	1.3	N/A

DETECTION CRITERION: 0.6

SODIUM

QUALITY CONTROL DATA FROM 03/01/84 TO 11/12/84

LAB: Domestic Water

Analytical Range: 0.4 to 200 mg/L as Na

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	98	130	129	-1	1.1
B :	98	33	33	0	0.6
A+B :	98	163	162	-0	1.3
A-B :	98	98	96	-1	1.2
C :	98	32.5	32.7	0.2	0.35
D :	98	6.5	6.4	-0.1	0.20
C+D :	98	39.0	39.1	0.1	0.43
C-D :	98	26.0	26.4	0.4	0.37

s.d(AB): Sw(within run): 0.8

S(between runs): 0.9

S/Sw: 1.04

s.d(CD): Sw(within run): 0.26

S(between runs): 0.28

S/Sw: 1.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

155 to 170 for A+B
93 to 103 for A-B
36.8 to 41.3 for C+D
24.5 to 27.5 for C-D

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
120	0.0 - 10.0	0.24	4.88
84	10.0 - 25.0	0.34	1.93
43	25.0 - 50.0	0.87	2.31
24	50.0 - 100	0.8	1.01
13	100 - 200	2.4	1.57
284	Overall	0.8	N/A

DETECTION CRITERION: 0.4

*** SODIUM ***

IDENTIFICATION:

Laboratory	: Precipitation	Method Introduced:	18/05/79
LIS Test Name Code:	NAUR	Units	: mg/L as Na
Work Station Code	: PRAA	Unit Code	: 064811
Method Code	: 002EA1	Supervisor	: M. Rawlings
Sample Type/Matrix: Precipitation, Throughfall, Stemflow.			

SAMPLING:

Quantity Required: 25 mL
Container : Polystyrene

ANALYTICAL PROCEDURE:

Samples are analysed by AAS at 589.0 nm with an air-acetylene flame. Potassium is added as a suppressant via an automated sampling train.
Approximate absorbance: 0.5 at the 1.00 mg/L level.

INSTRUMENTATION:

Automated modular continuous flow atomic absorption spectrophotometer (AAS) system

REPORTING:

Maximum Significant Figures: 3	
Minimum Increment (W) : 0.005	Detection Criterion (T): 0.02

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : BL plus 2 standards every 10 samples

SODIUM

QUALITY CONTROL DATA FROM 01/01/82 TO 25/12/82

LAB: Precipitation

Analytical Range: 0.02 to 1.00 mg/L as Na

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	99	0.60	0.60	-0.00	0.007
B :	98	0.10	0.10	-0.00	0.007
A+B :	98	0.70	0.70	-0.00	0.010
A-B :	98	0.50	0.50	0.00	0.009

s.d(AB): Sw(within run): 0.007 S(between runs): 0.007 S/Sw: 1.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.66 to 0.75 for A+B
0.47 to 0.53 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
71	0.00 - 0.20	0.012	11.52
14	0.20 - 0.50	0.006	1.77
6	0.50 - 1.00	0.013	1.77
91	Overall	0.011	N/A

DETECTION CRITERION: 0.02

SODIUM

QUALITY CONTROL DATA FROM 06/01/83 TO 29/12/83

LAB: Precipitation

Analytical Range: 0.01 to 1.00 mg/L as Na

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	48	0.60	0.60	0.00	0.010
B :	48	0.10	0.10	0.00	0.009
A+B :	48	0.70	0.71	0.01	0.016
A-B :	48	0.50	0.50	0.00	0.012

s.d(AB): Sw(within run): 0.008

S(between runs): 0.010

S/Sw: 1.19

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.66 to 0.75 for A+B
0.47 to 0.53 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
24	0.00 - 0.20	0.006	5.61
1	0.20 - 0.50	N/A	N/A
0	0.50 - 1.00	N/A	N/A
25	Overall	0.028	N/A

DETECTION CRITERION: 0.01

SODIUM

QUALITY CONTROL DATA FROM 04/01/84 TO 18/12/84

LAB: Precipitation

Analytical Range: 0.01 to 1.00 mg/L as Na

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	53	0.60	0.61	0.01	0.011
B :	53	0.10	0.10	-0.00	0.006
A+B :	53	0.70	0.71	0.01	0.012
A-B :	53	0.50	0.51	0.01	0.013

s.d(AB): Sw(within run): 0.009

S(between runs): 0.009

S/Sw: 0.98

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.66 to 0.75 for A+B
0.47 to 0.53 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
90	0.00 - 0.20	0.007	6.78
6	0.20 - 0.50	0.004	1.08
0	0.50 - 1.00	N/A	N/A
96	Overall	0.007	N/A

DETECTION CRITERION: 0.01

*** SODIUM ***

IDENTIFICATION:

Laboratory	: Rivers and Lakes	Method Introduced:	01/04/74
LIS Test Name Code:	NAUR	Units	: mg/L as Na
Work Station Code	: RMNAKH, RMNAKL	Unit Code	: 064811
Method Code	: 002CA1, 002DA1	Supervisor	: J. Crowther
Sample Type/Matrix:	Rivers, Lakes, Soil Extracts, Effluents.		

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm using an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train.
Approximate absorbance: 0.9 for both analytical ranges

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system(AAS).

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.05, 0.01 Detection Criterion (T): 0.1, 0.03

CALIBRATION:

BL plus 10 standards

CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA for each analytical range
Drift : BL plus 1 standard for each analytical range

MODIFICATIONS:

01/12/81- Calibration range became 10.0 mg/L full scale; second analytical range was dropped.
01/03/84- Analytical range(RMNAKL) was added; full scale:2.00 mg/L. This range is currently restricted to special programs.
01/09/84- Analytical range(RMNAKH) was increased from 10.0 to 20.0 mg/L full scale. Calibration technique was changed from quadratric to linear interpolation. Potassium is no longer determined simultaneously.

SODIUM

QUALITY CONTROL DATA FROM 15/01/82 TO 22/12/82

LAB: Rivers and Lakes

Analytical Range: 0.10 to 10.00 mg/L as Na

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	50	7.50	7.41	-0.09	0.072
B :	50	3.00	2.96	-0.04	0.060
A+B :	50	10.50	10.37	-0.13	0.103
A-B :	50	4.50	4.45	-0.05	0.083

s.d(AB): Sw(within run): 0.059 S(between runs): 0.066 S/Sw: 1.13

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.05 to 10.95 for A+B
4.20 to 4.80 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
125	0.00 - 2.00	0.063	6.35
17	2.00 - 5.00	0.067	1.93
11	5.00 - 10.00	0.120	1.61
153	Overall	0.070	N/A

DETECTION CRITERION: 0.10

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	27	0.00	0.000
Full Scale Abs :	49	0.666	0.1459

SODIUM

QUALITY CONTROL DATA FROM 07/01/83 TO 22/12/83

LAB: Rivers and Lakes

Analytical Range: 0.08 to 10.00 mg/L as Na

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	68	7.50	7.38	-0.12	0.115
B :	68	3.00	2.93	-0.07	0.057
A+B :	68	10.50	10.31	-0.19	0.133
A-B :	68	4.50	4.45	-0.05	0.124

s.d(AB): Sw(within run): 0.088 S(between runs): 0.091 S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.05 to 10.95 for A+B
4.20 to 4.80 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
209	0.00 - 2.00	0.049	4.85
22	2.00 - 5.00	0.083	2.36
4	5.00 - 10.00	0.048	0.64
235	Overall	0.053	N/A

DETECTION CRITERION: 0.08

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	67	0.00	0.000
Full Scale Abs :	65	0.978	0.3107

SODIUM

QUALITY CONTROL DATA FROM 05/01/84 TO 28/08/84

LAB: Rivers and Lakes

Analytical Range: 0.09 to 10.00 mg/L as Na

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	41	7.50	7.41	-0.09	0.133
B :	40	3.00	2.96	-0.04	0.081
A+B :	40	10.50	10.36	-0.14	0.177
A-B :	40	4.50	4.45	-0.05	0.129

s.d(AB): Sw(within run): 0.091 S(between runs): 0.110 S/Sw: 1.20

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.05 to 10.95 for A+B
4.20 to 4.80 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
101	0.00 - 2.00	0.054	5.39
12	2.00 - 5.00	0.045	1.29
7	5.00 - 10.00	0.095	1.27
120	Overall	0.056	N/A

DETECTION CRITERION: 0.09

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	40	0.00	0.005
Full Scale Abs :	41	0.733	0.1749

SODIUM

QUALITY CONTROL DATA FROM 27/02/84 TO 30/08/84

LAB: Rivers and Lakes

Analytical Range: 0.03 to 2.00 mg/L as Na

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	32	1.50	1.51	0.01	0.015
B :	32	0.60	0.61	0.01	0.013
A+B :	32	2.10	2.11	0.01	0.021
A-B :	32	0.90	0.90	0.00	0.018

s.d(AB): Sw(within run): 0.013

S(between runs): 0.014

S/Sw: 1.09

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.01 to 2.19 for A+B
0.84 to 0.96 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
0	0.00 - 0.20	N/A	N/A
66	0.20 - 0.80	0.018	3.62
43	0.80 - 2.00	0.024	1.73
109	Overall	0.021	N/A

DETECTION CRITERION: 0.03

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	32	0.00	0.000
High Std ABS :	32	0.762	0.2164

*** SOLIDS - DISSOLVED ***

IDENTIFICATION:

Laboratory	: Rivers and Lakes	Method Introduced:	01/04/74
LIS Test Name Code:	RSF	Units	: mg/L
Work Station Code	: RMTSD	Unit Code	: 064000
Method Code	: 101A15	Supervisor	: J. Crowther
Sample Type/Matrix:	Rivers, Lakes, Effluents		

SAMPLING:

Quantity Required: 75 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

Sample is filtered under moderate suction through a Whatman 934AH glass fibre filter. 50.0 mL of filtrate is pipetted into a preweighed ceramic dish, dried at 103 to 105 C, and stored in a desiccator until cool. After reweighing the dissolved residue or solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

INSTRUMENTATION:

-Balance(4/5-decimal places),drying oven, suction filtration apparatus, ceramic dishes
-Microcomputer system with appropriate software

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 2 Detection Criterion (T): Not applicable

CALIBRATION:

Balance zero and 1 built-in calibration weight

CONTROLS:

Calibration : 2 S class weights, eg, QCA
Recovery : LTBL plus 2 standards, eg, R1
Drift : Balance zero is checked frequently

MODIFICATIONS:

15/01/82- QC program was expanded to include recovery standards
01/05/84- Microcomputer control was introduced

NOTES:

As gravimetric analysis is rarely utilized for samples with low dissolved solids contents, sufficient data for calculating a detection criterion are not available. For such samples dissolved solids are estimated from conductivity. If then the conductivity of the sample is less than 400 uS/cm:

$$\text{Dissolved solids(mg/L)} = 0.65 \times \text{Conductivity(uS/cm)}$$

SOLIDS - DISSOLVED

QUALITY CONTROL DATA FROM 04/01/82 TO 30/12/82

LAB: Rivers and Lakes

Analytical Range:N/A to 1000 mg/L

CALIBRATION CONTROL: (g)

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av.Concn Measured</u>	<u>Av.Bias</u>	<u>Standard Deviation</u>
A :	143	50.0000	50.0004	0.0004	0.00019
B :	142	10.0000	9.9999	-0.0001	0.00023
A+B :	141	60.0000	60.0003	0.0003	0.00040
A-B :	141	40.0000	40.0005	0.0005	0.00015

s.d(AB): Sw(within run): 0.00011 S(between runs): 0.00021 S/Sw: 1.95

On any given day the calibration is accepted if the values obtained lie within the ranges:

59.9985 to 60.0015 for A+B
39.9990 to 40.0010 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av.Concn Measured</u>	<u>Standard Deviation</u>
R1 :	99	745.6	748.1	10.63
R2 :	97	279.6	284.3	10.22

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
0	0.0 - 50.0	N/A	N/A
0	50.0 - 100.0	N/A	N/A
8	100.0 - 250	6.7	3.82
184	250 - 500	10.4	2.77
57	500 - 1000	43.6	5.81
249	Overall	22.9	N/A

DETECTION CRITERION: N/A

SOLIDS - DISSOLVED

QUALITY CONTROL DATA FROM 04/01/83 TO 21/12/83

LAB: Rivers and Lakes

Analytical Range:N/A to 1000 mg/L

CALIBRATION CONTROL: (g)

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av.Concn Measured</u>	<u>Av.Bias</u>	<u>Standard Deviation</u>
A :	167	50.0000	50.0003	0.0003	0.00012
B :	167	10.0000	10.0000	-0.0000	0.00013
A+B :	166	60.0000	60.0003	0.0003	0.00020
A-B :	166	40.0000	40.0003	0.0003	0.00015

s.d(AB): Sw(within run): 0.00010 S(between runs): 0.00012 S/Sw: 1.17

On any given day the calibration is accepted if the values obtained lie within the ranges:

59.9985 to 60.0015 for A+B
39.9990 to 40.0010 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av.Concn Measured</u>	<u>Standard Deviation</u>
R1 :	128	745.6	747.0	18.23
R2 :	128	279.6	279.0	25.57

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
0	0.0 - 50.0	N/A	N/A
0	50.0 - 100.0	N/A	N/A
16	100.0 - 250	4.4	2.53
256	250 - 500	21.1	5.64
72	500 - 1000	24.3	3.24
344	Overall	21.5	N/A

DETECTION CRITERION: N/A

SOLIDS - DISSOLVED

QUALITY CONTROL DATA FROM 03/01/84 TO 18/12/84

LAB: Rivers and Lakes

Analytical Range: 1.4 to 1000 mg/L

CALIBRATION CONTROL: (g)

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	153	50.0000	50.0003	0.0003	0.00031
B :	153	10.0000	10.0000	0.0000	0.00010
A+B :	152	60.0000	60.0003	0.0003	0.00037
A-B :	152	40.0000	40.0002	0.0002	0.00027

s.d(AB): Sw(within run): 0.00019 S(between runs): 0.00023 S/Sw: 1.20

On any given day the calibration is accepted if the values obtained lie within the ranges:

59.9985 to 60.0015 for A+B
39.9990 to 40.0010 for A-B

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	132	745.6	749.3	23.62
R2 :	132	279.6	286.3	24.37

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
4	0.0 - 50.0	0.82	3.30
2	50.0 - 100.0	1.25	1.67
26	100.0 - 250	13.4	7.64
232	250 - 500	17.3	4.62
32	500 - 1000	10.9	1.45
296	Overall	16.3	N/A

DETECTION CRITERION: 1.4

*** SOLIDS - DISSOLVED ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: Before '61
LIS Test Name Code: RSF Units : mg/L
Work Station Code : SOLIDS Unit Code : 064000
Method Code : 101AI5 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,
Effluents

SAMPLING:

Quantity Required: 75 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

Sample is filtered under moderate suction through a Whatman 934AH glass fibre filter. 50.0 mL of filtrate is pipetted into a preweighed ceramic dish, dried at 103 to 105 C, and stored in a dessicator until cool. After reweighing the dissolved residue or solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

INSTRUMENTATION:

-Balance(4/5-decimal places),drying oven, suction filtration apparatus, ceramic dishes
Microcomputer system with appropriate software

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.5 Detection Criterion (T): 5

CALIBRATION:

Balance zero and 1 built-in calibration weight

CONTROLS:

Calibration : 2 S class weights, eg, QCA
Recovery : LTBL plus 2 standards, eg, R1
Drift : Balance zero is checked at least 4 times daily

MODIFICATIONS:

15/01/82- Microcomputer control was introduced

NOTES:

As the same two balances are used for all solids analyses in the Sewage/Industrial laboratory, the calibration control data are only listed once: in the Solids-Total report.

SOLIDS - DISSOLVED

QUALITY CONTROL DATA FROM 05/01/82 TO 22/10/82

LAB: Sewage/Industrial

Analytical Range: 8 to 3000 mg/L

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	20	1900	1887	33.6
R2 :	20	475	466	31.3

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
30	0 - 300	5.1	3.37
21	300 - 500	7.8	1.95
13	500 - 1000	12.5	1.67
10	1000 - 3000	12.2	0.61
74	Overall	8.6	N/A

DETECTION CRITERION: 8

SOLIDS - DISSOLVED

QUALITY CONTROL DATA FROM 01/01/83 TO 31/12/83

LAB: Sewage/Industrial

Analytical Range: 2 to 3000 mg/L

RECOVERIES:

		<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1	:	22	1900	1909	8.8
R2	:	22	475	478	5.6

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
4	0 - 500	1.0	0.40
9	500 - 1250	8.2	0.93
0	1250 - 3000	N/A	N/A
13	Overall	7.5	N/A

DETECTION CRITERION: 2

SOLIDS - DISSOLVED

QUALITY CONTROL DATA FROM 01/01/84 TO 31/12/84

LAB: Sewage/Industrial

Analytical Range: 2 to 3000 mg/L

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	15	1900	1903	10.5
R2 :	15	495	478	9.2

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
4	0 - 500	1.4	0.54
8	500 - 1250	0.8	0.09
0	1250 - 3000	N/A	N/A
12	Overall	0.3	N/A

DETECTION CRITERION: 2

*** SOLIDS - IGNITED ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: Before '61
LIS Test Name Code: RSFA,RSPA,RSTA Units : mg/L
Work Station Code : SOLIDS Unit Code : 064000
Method Code : 101AI5,201AI5,001AI5 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Domestic Waters, Leachate,
Effluents

SAMPLING:

Quantity Required: 75-500 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

The procedure for particulate, dissolved, or total solids is followed and the dried residue is ignited at 600 C for one hour in a muffle furnace. As soon as practical the dish is transferred to a desiccator to cool. The ignited or ash weight is obtained as the difference between the final ignited weight and the original dish weight. Similarly the volume used in the ignited calculations is the volume selected for the original dried solids measurement. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

INSTRUMENTATION:

-Balance(4/5 decimal places), muffle furnace, ceramic dishes, Petri dishes
-Microcomputer system with appropriate software

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.2,0.1,0.5 Detection Criterion (T): 8, 3, 30

CALIBRATION:

Balance zero and 1 built-in calibration weight

CONTROLS:

Calibration : 2 S class weights, eg, QCA
Drift : Balance zero is checked at least 4 times daily

MODIFICATIONS:

01/05/82 Microcomputer control was introduced

NOTES:

-In the order listed above, W and T values refer to the residual ash after ignition of the dried residual from dissolved, particulate, and total solids determinations.
-Duplicate data refer to ash residuals rather than loss on ignition.
-Detection criteria estimates are unreliable due to limited data; samples requiring these tests are usually sewage sludges with high solids contents.
-As the same two balances are used for all solids analyses in the Sewage/Industrial laboratory, the calibration control data are only listed once: in the Solids-Total report for Ignited Dissolved and Ignited Total tests, and in the Solids-Particulate report for Ignited Particulate tests.

SOLIDS - IGNITED

QUALITY CONTROL DATA FROM 06/01/82 TO 26/08/82

LAB: Sewage/Industrial

Analytical Range: - to 30000 mg/L

DUPLICATES: (Dissolved Ash)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
1	0 - 300	N/A	N/A
5	300 - 1000	10.7	1.65
2	1000 - 2500	9.9	0.57
1	2500 - 15000	N/A	N/A
0	15000 - 30000	N/A	N/A
9	Overall	8.8	N/A

DETECTION CRITERION: 18

DUPLICATES: (Particulate Ash)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
5	0.0 - 20.0	1.80	17.99
1	20.0 - 50.0	N/A	N/A
0	50.0 - 100.0	N/A	N/A
29	100.0 - 1000	22.0	4.00
28	1000 - 3000	40.7	2.04
63	Overall	34.6	N/A

DETECTION CRITERION: 3.0

DUPLICATES: (Total Ash)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
1	0 - 300	N/A	N/A
3	300 - 1000	20.5	3.16
3	1000 - 2500	10.1	0.58
17	2500 - 15000	145.3	1.66
13	15000 - 30000	1088.5	4.84
37	Overall	674.1	N/A

DETECTION CRITERION: N/A

SOLIDS - IGNITED

QUALITY CONTROL DATA FROM 01/01/83 TO 31/12/83

LAB: Sewage/Industrial

Analytical Range: - to 30000 mg/L

DUPLICATES: (Dissolved Ash)

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
4	0 - 500	3.7	1.47
7	500 - 1250	9.5	1.09
0	1250 - 30000	N/A	N/A
11	Overall	8.0	N/A

DETECTION CRITERION: 6

DUPLICATES: (Particulate Ash)

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
9	0 - 500	5.5	2.20
12	500 - 1250	11.5	1.32
13	1250 - 3000	18.7	0.88
34	Overall	15.0	N/A

DETECTION CRITERION: 9

DUPLICATES: (Total Ash)

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
0	0 - 500	N/A	N/A
5	500 - 1250	17.6	2.01
12	1250 - 6000	15.0	0.41
7	6000 - 15000	42.4	0.40
7	15000 - 30000	212.9	0.95
31	Overall	138.4	N/A

DETECTION CRITERION: 29

SOLIDS - IGNITED

QUALITY CONTROL DATA FROM 01/01/84 TO 31/12/84

LAB: Sewage/Industrial

Analytical Range: - to 30000 mg/L

DUPLICATES: (Dissolved Ash)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
1	0 - 500	N/A	N/A
3	500 - 1250	5.1	0.58
0	1250 - 30000	N/A	N/A
4	Overall	2.6	N/A

DETECTION CRITERION: 8

DUPLICATES: (Particulate Ash)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
3	0 - 500	6.2	2.48
5	500 - 1250	11.0	1.26
9	1250 - 3000	12.2	0.57
17	Overall	12.3	N/A

DETECTION CRITERION: 10

DUPLICATES: (Total Ash)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
1	0 - 500	N/A	N/A
3	500 - 1250	13.8	1.58
8	1250 - 6000	20.2	0.56
3	6000 - 15000	33.5	0.32
7	15000 - 30000	252.8	1.12
22	Overall	151.3	N/A

DETECTION CRITERION: 23

*** SOLIDS - PARTICULATE ***

IDENTIFICATION:

Laboratory	: Rivers and Lakes	Method Introduced:	01/04/74
LIS Test Name Code:	RSP	Units	: mg/L
Work Station Code	: RMTSD	Unit Code	: 064000
Method Code	: 202A16	Supervisor	: J. Crowther
Sample Type/Matrix:	Rivers, Lakes, Effluents		

SAMPLING:

Quantity Required: 150-500 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

An appropriate sample volume (100 to 500 mL) is quickly poured into a graduated cylinder, and the volume is measured. The aliquot is then filtered under moderate suction through a prewashed and preweighed Whatman 934AH glass fibre filter. The cylinder and then the filter are washed with 25 mL distilled water; the rinse step is repeated. The filter is dried at 103 to 105 C, and stored in a desiccator until cool. After reweighing, the particulate residue or suspended solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

INSTRUMENTATION:

-Balance(6-decimal places), drying oven, suction filtration apparatus
-Microcomputer system with appropriate software

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.01 Detection Criterion (T): 0.7

CALIBRATION:

Balance zero and 1 built-in calibration weight

CONTROLS:

Calibration : 2 S class weights, eg, QCA
Recovery : LTBL plus 2 standards, eg, R1
Drift : Balance zero is checked frequently

MODIFICATIONS:

01/03/84- QC program was expanded to include recovery standards.
01/05/84- Microcomputer control was introduced.

NOTES:

In the following data, "Dry Filt Weight" is the change in weight of a tared filter paper after drying. "Washed Filt Wt" is the change in weight of a tared filter paper that was used to filter 200 mL of distilled, deionized water and then reweighed.

SOLIDS - PARTICULATE

QUALITY CONTROL DATA FROM 05/01/82 TO 29/12/82

LAB: Rivers and Lakes

Analytical Range: 0.7 to 1000 mg/L

CALIBRATION CONTROL (mg):

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	191	99.97	99.97	0.00	0.007
B :	191	19.90	19.90	-0.00	0.014
A+B :	190	119.87	119.87	0.00	0.018
A-B :	190	80.07	80.07	0.00	0.014

s.d(AB): Sw(within run): 0.010 S(between runs): 0.011 S/Sw: 1.17

On any given day the calibration is accepted if the values obtained lie within the ranges:

119.80 to 119.95 for A+B
80.02 to 80.12 for A-B

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
45	0.0 - 2.0	0.44	44.00
67	2.0 - 5.0	0.32	9.12
139	5.0 - 25.0	0.79	5.25
72	25.0 - 100	4.2	6.72
14	100 - 1000	7.1	1.29
337	Overall	3.2	N/A

DETECTION CRITERION: 0.7

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Dry Filt Weight :	128	-0.04	0.077
Washed Filt Wt :	129	0.01	0.221

SOLIDS - PARTICULATE

QUALITY CONTROL DATA FROM 04/01/83 TO 21/12/83

LAB: Rivers and Lakes

Analytical Range: 0.3 to 1000 mg/L

CALIBRATION CONTROL (mg):

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	178	99.97	99.97	0.00	0.004
B :	178	19.90	19.90	0.00	0.008
A+B :	177	119.87	119.88	0.01	0.010
A-B :	177	80.07	80.07	-0.00	0.008

s.d(AB): Sw(within run): 0.006

S(between runs): 0.007

S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

119.80 to 119.95 for A+B
80.02 to 80.12 for A-B

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
24	0.0 - 2.0	0.16	16.02
73	2.0 - 5.0	0.32	9.19
171	5.0 - 25.0	1.25	8.35
91	25.0 - 100	3.9	6.19
9	100 - 1000	6.6	1.19
368	Overall	2.5	N/A

DETECTION CRITERION: 0.3

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Dry Filt Weight :	136	-0.08	0.057
Washed Filt Wt :	137	-0.10	0.348

SOLIDS - PARTICULATE

QUALITY CONTROL DATA FROM 03/01/84 TO 28/12/84

LAB: Rivers and Lakes

Analytical Range: 0.7 to 1000 mg/L

CALIBRATION CONTROL (mg):

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	155	99.97	99.97	0.00	0.004
B :	155	19.90	19.90	0.00	0.007
A+B :	154	119.87	119.88	0.01	0.008
A-B :	154	80.07	80.07	-0.00	0.007

s.d(AB): Sw(within run): 0.005 S(between runs): 0.005 S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

119.80 to 119.95 for A+B
80.02 to 80.12 for A-B

RECOVERIES:

	Number of Data	Expected Concn	Av. Concn Measured	Standard Deviation
R1 :	131	50.00	49.33	2.199
R2 :	132	10.00	9.71	1.353

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
27	0.0 - 2.0	0.45	45.06
62	2.0 - 5.0	1.45	41.57
162	5.0 - 25.0	1.63	10.89
45	25.0 - 100	5.4	8.60
13	100 - 1000	17.5	3.18
309	Overall	5.0	N/A

DETECTION CRITERION: 0.7

OTHER CHECKS:

	Number of Data	Data Mean	Standard Deviation
Washed Filt Wt :	54	0.43	0.842

*** SOLIDS - PARTICULATE ***

IDENTIFICATION:

Laboratory : Sewage/Industrial Method Introduced: Before '61
LIS Test Name Code: RSP Units : mg/L
Work Station Code : SOLIDS Unit Code : 064
Method Code : 201A15 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Drinking Waters, Leachate, Effluents

SAMPLING:

Quantity Required: 75-500 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

An appropriate sample volume (50 to 500 mL) is quickly poured into a graduated cylinder, and the volume is measured. The aliquot is then filtered under moderate suction through a preweighed Whatman 934AH glass fibre filter. The cylinder and then the filter are washed with 50 mL distilled water. The filter is dried at 103 to 105 C, and stored in a desiccator until cool. After reweighing, the particulate residue or suspended solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

INSTRUMENTATION:

-Balance (5-decimal places), drying oven, suction filtration apparatus
-Microcomputer system with appropriate software

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.1 Detection Criterion (T): 1

CALIBRATION:

Balance zero and 1 built-in calibration weight

CONTROLS:

Calibration : 2 S class weights, eg, QCA for each balance
Recovery : LTBL plus 2 standards, eg, R1
Drift : Balance zero is checked at least 4 times daily

MODIFICATIONS:

01/07/81- Current microcomputer control system was introduced.
01/03/83- QC program was expanded to include recovery standards.
01/05/83 - Prerinsing of filters was discontinued. Instead, 5 filters from each box of 100 are weighed before and after rinsing to correct results for filters used with samples.
01/07/83 - New glass and acrylic filter holders (Whatman 90 mm) replaced Buchner funnels. Size and position of pores in the two types of holders is similar, but the filtration area is smaller and the seal at the filter edge is superior with the new holder.

SOLIDS - PARTICULATE

QUALITY CONTROL DATA FROM 15/01/82 TO 29/11/82

LAB: Sewage/Industrial

Analytical Range: 1 to 3000 mg/L

CALIBRATION CONTROL: (g)

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	201	0.50000	0.50005	0.00005	0.000027
B :	201	0.05000	0.05003	0.00003	0.000032
A+B :	200	0.55000	0.55008	0.00008	0.000052
A-B :	200	0.45000	0.45003	0.00003	0.000028
C :	188	0.50000	0.50004	0.00004	0.000031
D :	188	0.05000	0.05002	0.00002	0.000038
C+D :	187	0.55000	0.55006	0.00006	0.000060
C-D :	187	0.45000	0.45001	0.00001	0.000035

s.d(AB): Sw(within run): 0.000020 S(between runs): 0.000030 S/Sw: 1.50
s.d(CD): Sw(within run): 0.000025 S(between runs): 0.000035 S/Sw: 1.39

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.54985 to 0.55015 for A+B
0.44990 to 0.45010 for A-B
0.54985 to 0.55015 for C+D
0.44990 to 0.45010 for C-D

RECOVERIES:

	Number of Data	Expected Concn	Av. Concn Measured	Standard Deviation
R1 :	16	187	169	8.2
R2 :	16	47	42	1.9

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
7	0 - 20	0.8	8.45
8	20 - 50	4.6	13.12
14	50 - 100	2.3	3.05
35	100 - 1000	20.5	3.72
22	1000 - 3000	80.2	4.01
86	Overall	44.9	N/A

DETECTION CRITERION: 1

SOLIDS - PARTICULATE

QUALITY CONTROL DATA FROM 01/01/83 TO 31/12/83

LAB: Sewage/Industrial

Analytical Range: 1 to 3000 mg/L

CALIBRATION CONTROL: (g)

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	106	0.50000	0.50004	0.00004	0.000031
B :	106	0.05000	0.05001	0.00001	0.000031
A+B :	106	0.55000	0.55005	0.00005	0.000030
A-B :	106	0.45000	0.45003	0.00003	0.000026
C :	135	0.50000	0.50002	0.00002	0.000033
D :	135	0.05000	0.05002	0.00002	0.000034
C+D :	135	0.55000	0.55004	0.00004	0.000036
C-D :	135	0.45000	0.45000	0.00000	0.000013

s.d(AB): Sw(within run): 0.000018 S(between runs): 0.000031 S/Sw: 1.71
s.d(CD): Sw(within run): 0.000009 S(between runs): 0.000033 S/Sw: 3.68

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.54985 to 0.55015 for A+B
0.44990 to 0.45010 for A-B
0.54985 to 0.55015 for C+D
0.44990 to 0.45010 for C-D

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	40	187	181	4.2
R2 :	42	47	45	2.8

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
15	0 - 20	0.7	7.00
25	20 - 50	0.8	2.29
50	50 - 100	2.4	3.20
52	100 - 1250	30.5	4.52
11	1250 - 3000	35.0	1.65
153	Overall	16.4	N/A

DETECTION CRITERION: 1

SOLIDS - PARTICULATE

QUALITY CONTROL DATA FROM 01/01/84 TO 31/12/84

LAB: Sewage/Industrial

Analytical Range: 1 to 3000 mg/L

CALIBRATION CONTROL: (g)

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	71	0.50000	0.50000	0.00000	0.000028
B :	71	0.05000	0.05000	0.00000	0.000023
A+B :	71	0.55000	0.55000	0.00000	0.000046
A-B :	71	0.45000	0.45000	0.00000	0.000023
C :	80	0.50000	0.50002	0.00002	0.000039
D :	80	0.05000	0.05002	0.00002	0.000035
C+D :	80	0.55000	0.55004	0.00004	0.000067
C-D :	80	0.45000	0.45000	0.00000	0.000030

s.d(AB): Sw(within run): 0.000016 S(between runs): 0.000026 S/Sw: 1.58
s.d(CD): Sw(within run): 0.000021 S(between runs): 0.000037 S/Sw: 1.75

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.54985 to 0.55015 for A+B
0.44990 to 0.45010 for A-B
0.54985 to 0.55015 for C+D
0.44990 to 0.45010 for C-D

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	36	187	183	1.9
R2 :	36	47	46	1.7

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
21	0 - 20	0.7	7.00
14	20 - 50	1.1	3.14
22	50 - 100	1.8	2.40
28	100 - 1250	9.0	1.33
9	1250 - 3000	40.2	1.89
94	Overall	15.0	N/A

DETECTION CRITERION: 1

*** SOLIDS- TOTAL ***

IDENTIFICATION:

Laboratory	: Sewage/Industrial	Method Introduced:	Before '61
LIS Test Name Code:	RST	Units	: mg/L
Work Station Code	: SOLIDS	Unit Code	: 064000
Method Code	: 001A15	Supervisor	: P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters, Effluents, Sludge			

SAMPLING:

Quantity Required: 75 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

A 50.0 mL aliquot of sample is pipetted into a preweighed ceramic dish, dried at 103 to 105 C, and stored in a desiccator until cool. After reweighing the total residue or solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

INSTRUMENTATION:

-Balance(4/5-decimal places),drying oven, ceramic dishes
-Microcomputer system with appropriate software

REPORTING:

Maximum Significant Figures: 3	
Minimum Increment (W) : 0.5	Detection Criterion (T): 20

CALIBRATION:

Balance zero and 1 built-in calibration weight

CONTROLS:

Calibration : 2 S class weights, eg, QCA
Recovery : BL plus 2 standards, eg, R1
Drift : Balance zero is checked at least 4 times daily

MODIFICATIONS:

15/01/82 Microcomputer control was introduced

SOLIDS - TOTAL

QUALITY CONTROL DATA FROM 06/01 82 TO 29/11/82

LAB: Sewage/Industrial

Analytical Range: 17 to 30000 mg/L

CALIBRATION CONTROL: (g)

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	209	50.00000	50.00011	0.00011	0.000056
B :	209	30.00000	29.99990	-0.00010	0.000055
A+B :	208	80.00000	80.00002	0.00002	0.000115
A-B :	208	20.00000	20.00021	0.00021	0.000056
C :	196	50.00000	50.00010	0.00010	0.000079
D :	196	30.00000	29.99990	-0.00010	0.000046
C+D :	194	80.00000	80.00001	0.00001	0.000099
C-D :	194	20.00000	20.00019	0.00019	0.000050

s.d(AB): Sw(within run): 0.000040 S(between runs): 0.000055 S/Sw: 1.39
s.d(CD): Sw(within run): 0.000035 S(between runs): 0.000065 S/Sw: 1.85

On any given day the calibration is accepted if the values obtained lie within the ranges:

79.99985 to 80.00015 for A+B
19.99990 to 20.00010 for A-B
79.99985 to 80.00015 for C+D
19.99990 to 20.00010 for C-D

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	24	19000	18751	243.5
R2 :	24	1900	1890	32.5

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
12	0 - 300	10.5	6.98
17	300 - 1000	10.4	1.59
10	1000 - 2500	9.7	0.55
34	2500 - 15000	121.9	1.39
23	15000 - 30000	117.0	0.52
96	Overall	108.5	N/A

DETECTION CRITERION: 17

SOLIDS - TOTAL

QUALITY CONTROL DATA FROM 01/01/83 TO 31/12/83

LAB: Sewage/Industrial

Analytical Range: 99 to 30000 mg/L

CALIBRATION CONTROL: (g)

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	106	50.00000	50.00009	0.00009	0.000046
B :	106	30.00000	29.99987	-0.00013	0.000051
A+B :	106	80.00000	79.99996	-0.00004	0.000163
A-B :	106	20.00000	20.00021	0.00021	0.000234
C :	135	50.00000	50.00010	0.00010	0.000057
D :	135	30.00000	29.99989	-0.00011	0.000050
C+D :	135	80.00000	79.99999	-0.00001	0.000167
C-D :	135	20.00000	20.00021	0.00021	0.000215

s.d(AB): Sw(within run): 0.000165 S(between runs): 0.000049 S/Sw: 0.29

s.d(CD): Sw(within run): 0.000152 S(between runs): 0.000053 S/Sw: 0.35

On any given day the calibration is accepted if the values obtained lie within the ranges:

79.99900	to	80.00100	for A+B
19.99933	to	20.00067	for A-B
79.99900	to	80.00100	for C+D
19.99933	to	20.00067	for C-D

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	28	19000	18983	83.6
R2 :	28	1900	1907	10.9

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
17	0 - 6000	60.2	2.01
6	6000 - 15000	107.6	1.02
7	15000 - 30000	273.7	1.22
30	Overall	152.9	N/A

DETECTION CRITERION: 99

SOLIDS - TOTAL

QUALITY CONTROL DATA FROM 01/01/84 TO 31/12/84

LAB: Sewage/Industrial

Analytical Range: 39 to 30000 mg/L

CALIBRATION CONTROL: (g)

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	71	50.00000	50.00035	0.00035	0.000172
B :	71	30.00000	30.00004	0.00004	0.000121
A+B :	71	80.00000	80.00039	0.00039	0.000272
A-B :	71	20.00000	20.00031	0.00031	0.000120
C :	80	50.00000	50.00012	0.00012	0.000066
D :	80	30.00000	29.99990	-0.00010	0.000051
C+D :	80	80.00000	80.00001	0.00001	0.000108
C-D :	80	20.00000	20.00022	0.00022	0.000046

s.d(AB): Sw(within run): 0.000085 S(between runs): 0.000149 S/Sw: 1.75
s.d(CD): Sw(within run): 0.000033 S(between runs): 0.000059 S/Sw: 1.81

On any given day the calibration is accepted if the values obtained lie within the ranges:

79.99900 to 80.00100 for A+B
19.99933 to 20.00067 for A-B
79.99900 to 80.00100 for C+D
19.99933 to 20.00067 for C-D

RECOVERIES:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Standard Deviation</u>
R1 :	16	19000	19095	61.2
R2 :	16	1900	1905	12.9

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
9	0 - 6000	23.7	0.79
6	6000 - 15000	36.7	0.35
12	15000 - 30000	152.4	0.68
27	Overall	123.3	N/A

DETECTION CRITERION: 39

*** SULPHATE ***

IDENTIFICATION:

Laboratory	: Domestic Water	Method Introduced:	Before '75
LIS Test Name Code:	SS04UR	Units	: mg/L as SO ₄
Work Station Code	: WS04	Unit Code	: 064941
Method Code	: 003AC1	Supervisor	: M. Rawlings
Sample Type/Matrix: Domestic Waters, Leachates, Effluents			

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

Sample is pumped through an ion exchange column to suppress cationic interferences, and then the sulphate reacts with barium methyl-thymol blue to produce barium sulphate and methyl-thymol blue (MTB). The absorbance of MTB provides a measurement of the sulphate concentration.
Approximate absorbance: 1.0 at the 100 mg/L level.

INSTRUMENTATION:

Basic automated modular continuous flow system plus the modules: 37 C heating bath (7.7 mL delay), cationic exchange column. Colourimetric measurement is through a 55.0 cm light path at 460 nm.

REPORTING:

Maximum Significant Figures: 3	
Minimum Increment (W) : 0.5	Detection Criterion (T): 1.6

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : BL plus 2 standards
Interference: BL spiked with 50 mg/L magnesium SO₄ and 200 mg/L calcium confirms efficiency of cation removal.

MODIFICATIONS:

01/10/81- A carbon dioxide trap was installed on the sodium hydroxide reagent.
01/10/84- A heating bath module (37 C) was installed preceeding the colourimeter.

SULPHATE

QUALITY CONTROL DATA FROM 05/01/82 TO 22/12/82

LAB: Domestic Water

Analytical Range: 1.2 to 100.0 mg/L as SO₄

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	94	58.0	58.2	0.2	1.00
B :	94	11.6	11.5	-0.1	0.67
A+B :	93	69.6	69.7	0.1	1.41
A-B :	93	46.4	46.7	0.3	0.96

s.d(AB): Sw(within run): 0.68 S(between runs): 0.85 S/Sw: 1.25

On any given day the calibration is accepted if the values obtained lie within the ranges:

65.1 to 74.1 for A+B
43.4 to 49.4 for A-B

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
69	0.0 - 20.0	0.74	7.37
128	20.0 - 50.0	0.68	1.94
41	50.0 - 100.0	1.11	1.48
238	Overall	0.79	N/A

DETECTION CRITERION: 1.2

SULPHATE

QUALITY CONTROL DATA FROM 06/01/83 TO 30/12/83

LAB: Domestic Water

Analytical Range: 0.6 to 100.0 mg/L as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	70	58.0	58.0	0.0	0.86
B :	70	11.6	12.1	0.5	0.53
A+B :	70	69.6	70.1	0.5	1.19
A-B :	70	46.4	46.0	-0.4	0.80

s.d(AB): Sw(within run): 0.56

S(between runs): 0.72

S/Sw: 1.27

On any given day the calibration is accepted if the values obtained lie within the ranges:

65.1 to 74.1 for A+B

43.4 to 49.4 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
73	0.0 - 20.0	0.34	3.37
89	20.0 - 50.0	0.66	1.89
30	50.0 - 100.0	0.49	0.65
192	Overall	0.54	N/A

DETECTION CRITERION: 0.6

SULPHATE

QUALITY CONTROL DATA FROM 06/01/84 TO 14/12/84

LAB: Domestic Water

Analytical Range: 1.6 to 100.0 mg/L as SO₄

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	81	58.0	58.0	-0.0	0.96
B :	81	11.6	11.9	0.3	0.45
A+B :	81	69.6	69.9	0.3	1.23
A-B :	81	46.4	46.1	-0.3	0.86

s.d(AB): Sw(within run): 0.61 S(between runs): 0.75 S/Sw: 1.24

On any given day the calibration is accepted if the values obtained lie within the ranges:

65.1 to 74.1 for A+B
43.4 to 49.4 for A-B

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
91	0.0 - 20.0	0.95	9.49
98	20.0 - 50.0	1.14	3.24
31	50.0 - 100.0	2.66	3.55
220	Overall	1.39	N/A

DETECTION CRITERION: 1.6

*** SULPHATE - PRECIPITATION ***

IDENTIFICATION:

Laboratory	: Precipitation	Method Introduced:	01/04/78
LIS Test Name Code:	SS04UR	Units	: mg/L as SO ₄
Work Station Code	: PRIC1	Unit Code	: 064941
Method Code	: 003AIO	Supervisor	: M. Rawlings
Sample Type/Matrix: Precipitation, Throughfall, Stemflow.			

SAMPLING:

Quantity Required: 15 mL
Container : Polystyrene

ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO₄ is determined by comparison of the sample scan to a series of standard scans.
Full scale conductivity : 10 uS/cm.
Nitrate and chloride are determined simultaneously.

INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.05 Detection Criterion (T): 0.07

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : 1 standard every 10 samples

SULPHATE

QUALITY CONTROL DATA FROM 06/01/82 TO 24/12/82

LAB: Precipitation

Analytical Range: 0.07 to 10.00 mg/L as SO₄

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	241	8.00	7.99	-0.01	0.077
B :	242	2.00	1.98	-0.02	0.038
A+B :	239	10.00	9.97	-0.03	0.089
A-B :	239	6.00	6.01	0.01	0.082

s.d(AB): Sw(within run): 0.058 S(between runs): 0.060 S/Sw: 1.04

On any given day the calibration is accepted if the values obtained lie within the ranges:

9.55 to 10.45 for A+B
5.70 to 6.30 for A-B

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
90	0.00 - 2.00	0.043	4.26
120	2.00 - 5.00	0.083	2.37
60	5.00 - 10.00	0.100	1.33
270	Overall	0.081	N/A

DETECTION CRITERION: 0.07

SULPHATE

QUALITY CONTROL DATA FROM 04/01/83 TO 22/12/83

LAB: Precipitation

Analytical Range: 0.07 to 10.00 mg/L as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	317	8.00	7.96	-0.04	0.082
B :	317	2.00	2.01	0.01	0.038
A+B :	316	10.00	9.97	-0.03	0.089
A-B :	316	6.00	5.96	-0.04	0.091

s.d(AB): Sw(within run): 0.064

S(between runs): 0.064

S/Sw: 0.99

On any given day the calibration is accepted if the values obtained lie within the ranges:

9.55 to 10.45 for A+B

5.70 to 6.30 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
96	0.00 - 2.00	0.044	4.44
119	2.00 - 5.00	0.064	1.81
52	5.00 - 10.00	0.116	1.54
267	Overall	0.073	N/A

DETECTION CRITERION: 0.07

SULPHATE

QUALITY CONTROL DATA FROM 09/01/84 TO 20/12/84

LAB: Precipitation

Analytical Range: 0.06 to 10.00 mg/L as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	170	8.00	8.00	-0.00	0.085
B :	170	2.00	2.01	0.01	0.039
A+B :	169	10.00	10.01	0.01	0.100
A-B :	169	6.00	5.99	-0.01	0.088

s.d(AB): Sw(within run): 0.062 S(between runs): 0.066 S/Sw: 1.07

On any given day the calibration is accepted if the values obtained lie within the ranges:

9.55 to 10.45 for A+B
5.70 to 6.30 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
103	0.00 - 2.00	0.039	3.91
123	2.00 - 5.00	0.073	2.08
53	5.00 - 10.00	0.107	1.43
279	Overall	0.074	N/A

DETECTION CRITERION: 0.06

*** SULPHATE ***

IDENTIFICATION:

Laboratory	: Precipitation	Method Introduced:	01/07/80
LIS Test Name Code:	SS04FR SS04NF	Units	: ug/Filter as SO4
Work Station Code	: PRSEQ	Unit Code	: 361941
Method Code	: 004AI0	Supervisor	: M. Rawlings

Sample Type/Matrix: Teflon and nylon filters from sequential filter packs and nylon filters from LoVol filter packs.

SAMPLING:

Quantity Required: 1 filter
Container : Polyethylene bag

SAMPLE PREPARATION:

Filters are extracted with 25 ml. of DDW (teflon) or 25 ml. of .03N NaOH (nylon) in polystyrene tubes with ultrasonic treatment followed by a 24 hour rest period.

ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO4 is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as SO4.
Full scale conductivity : 30 uS/cm.
Nitrate and chloride are determined simultaneously.

INSTRUMENTATION:

- Ultrasonic bath; polystyrene tubes
- Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 1.25 ug/filter Detection Criterion (T): 2 ug/filter

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : 1 standard every 10 samples

MODIFICATIONS:

01/07/80 - Ion chromatographic procedure for precipitation samples was modified for analysis of teflon and nylon filter extracts by developing the above filter extraction procedure
10/03/84 - Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced.

NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

SULPHATE

QUALITY CONTROL DATA FROM 06/01/82 TO 03/12/82

LAB: Precipitation

Analytical Range: 3.9 to 250.0 ug/filter as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	138	200.0	200.2	0.2	2.68
B :	141	50.0	50.7	0.7	1.13
A+B :	137	250.0	250.9	0.9	2.95
A-B :	137	150.0	149.5	-0.5	2.86

s.d(AB): Sw(within run): 2.02

S(between runs): 2.06

S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

238.8 to 261.3 for A+B
142.5 to 157.5 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
62	0.0 - 50.0	2.37	9.47
6	50.0 - 125.0	2.74	3.13
0	125.0 - 250.0	N/A	N/A
68	Overall	2.39	N/A

DETECTION CRITERION: 3.9

SULPHATE

QUALITY CONTROL DATA FROM 04/01/83 TO 22/12/83

LAB: Precipitation

Analytical Range: 1.5 to 250.0 ug/filter as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	85	200.0	199.3	-0.7	2.19
B :	85	50.0	51.2	1.2	2.27
A+B :	85	250.0	250.5	0.5	3.57
A-B :	85	150.0	148.1	-1.9	2.66

s.d(AB): Sw(within run): 1.88

S(between runs): 2.23

S/Sw: 1.18

On any given day the calibration is accepted if the values obtained lie within the ranges:

238.8 to 261.3 for A+B

142.5 to 157.5 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
29	0.0 - 50.0	0.91	3.62
8	50.0 - 125.0	10.55	12.06
2	125.0 - 250.0	3.54	1.89
39	Overall	5.24	N/A

DETECTION CRITERION: 1.5

SULPHATE

QUALITY CONTROL DATA FROM 03/01/84 TO 27/12/84

LAB: Precipitation

Analytical Range: 2.3 to 250.0 ug/filter as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	136	200.0	201.0	1.0	2.43
B :	136	50.0	51.0	1.0	1.33
A+B :	135	250.0	252.0	2.0	2.84
A-B :	135	150.0	149.9	-0.1	2.69

s.d(AB): Sw(within run): 1.90

S(between runs): 1.96

S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

238.8 to 261.3 for A+B
142.5 to 157.5 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
113	0.0 - 50.0	1.42	5.69
17	50.0 - 125.0	5.21	5.96
7	125.0 - 250.0	4.60	2.46
137	Overall	2.52	N/A

DETECTION CRITERION: 2.3

*** SULPHATE ***

IDENTIFICATION:

Laboratory	: Precipitation	Method Introduced:	01/07/80
LIS Test Name Code:	SS04UR	Units	: ug/Filter as SO4
Work Station Code	: PRLDV	Unit Code	: 361941
Method Code	: 004AIC	Supervisor	: M. Rawlings
Sample Type/Matrix: W40 filters from LoVol filter packs.			

SAMPLING:

Quantity Required: 1 filter
Container : Polyethylene bags

SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polyethylene tubes with ultrasonic treatment followed by a 24 hour rest period.

ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na₂CO₃/NaHCO₃ to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO₄ is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as SO₄.

Full scale conductivity : 30 uS/cm.

N.B. Nitrate and chloride are determined simultaneously.

INSTRUMENTATION:

- Ultrasonic bath; polyethylene tubes
- Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 2.50 ug/filter Detection Criterion (T): 5 ug/filter

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : 1 standard every 10 samples

MODIFICATIONS:

01/08/81 - Ion chromatographic procedure for precipitation samples was modified for analysis of LoVol W40 filter extracts by developing the above filter extraction procedure.

10/03/84 - Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na₂CO₃/NaHCO₃ was introduced.

NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

SULPHATE

QUALITY CONTROL DATA FROM 06/01/82 TO 03/12/82

LAB: Precipitation

Analytical Range: 7.8 to 500.0 ug/filter as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	138	400.0	400.4	0.4	5.36
B :	141	100.0	101.4	1.4	2.26
A+B :	137	500.0	501.9	1.9	5.90
A-B :	137	300.0	299.0	-1.0	5.72

s.d(AB): Sw(within run): 4.04

S(between runs): 4.11

S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

477.5 to 522.5 for A+B
285.0 to 315.0 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
62	0.0 - 100.0	4.74	9.47
6	100.0 - 250.0	5.48	3.13
0	250.0 - 500.0	N/A	N/A
68	Overall	4.78	N/A

DETECTION CRITERION: 7.8

SULPHATE

QUALITY CONTROL DATA FROM 04/01/83 TO 22/12/83

LAB: Precipitation

Analytical Range: 3.0 to 500.0 ug/filter as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	85	400.0	398.6	-1.4	4.37
B :	85	100.0	102.4	2.4	4.54
A+B :	85	500.0	501.0	1.0	7.15
A-B :	85	300.0	296.2	-3.8	5.32

s.d(AB): Sw(within run): 3.76

S(between runs): 4.45

S/Sw: 1.18

On any given day the calibration is accepted if the values obtained lie within the ranges:

477.5 to 522.5 for A+B
285.0 to 315.0 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
29	0.0 - 100.0	1.81	3.62
8	100.0 - 250.0	21.11	12.06
2	250.0 - 500.0	7.07	1.89
39	Overall	10.47	N/A

DETECTION CRITERION: 3.0

SULPHATE

QUALITY CONTROL DATA FROM 03/01/84 TO 27/12/84

LAB: Precipitation

Analytical Range: 4.7 to 500.0 ug/filter as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	136	400.0	401.9	1.9	4.85
B :	136	100.0	102.1	2.1	2.66
A+B :	135	500.0	504.0	4.0	5.68
A-B :	135	300.0	299.9	-0.1	5.39

s.d(AB): Sw(within run): 3.81

S(between runs): 3.91

S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

477.5 to 522.5 for A+B
285.0 to 315.0 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
113	0.0 - 100.0	2.84	5.69
17	100.0 - 250.0	10.43	5.96
7	250.0 - 500.0	9.21	2.46
137	Overall	5.05	N/A

DETECTION CRITERION: 4.7

*** SULPHATE ***

IDENTIFICATION:

Laboratory	: Rivers and Lakes	Method Introduced:	01/04/82
LIS Test Name Code:	SS04UR	Units	: mg/L as SO ₄
Work Station Code	: RMDSO4	Unit Code	: 064941
Method Code	: 003AI0	Supervisor	: J. Crowther
Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents			

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of sulphate in mg/L as SO₄ is determined by comparison of the sample scan to a series of standard scans.

Full scale conductivity : 30 uS/cm.

INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus control module(in-house design) for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3	
Minimum Increment (W) : 0.1	Detection Criterion (T): 0.2

CALIBRATION:

BL plus 10 standards

CONTROLS:

Calibration : LTBL plus 4 standards, eg, QCA
Drift : BL plus 2 standards

MODIFICATIONS:

01/04/82- The sulphate procedure that was introduced differed slightly from Method B in HAMES: (1) full scale values for the analytical ranges were 20.0 and 50.0 mg/L, and (2) samples were not spiked with concentrated eluent. The latter was not necessary because only sulphate was measured and spiking is required for chloride analysis.

01/01/84- Packed suppressor column was replaced by a fibre suppressor (walls of fibre are ion-exchange media). Full scale for high analytical was increased from 50.0 to 100 mg/L as SO₄; QC standards were adjusted accordingly. Analytical rate was doubled.

SULPHATE

QUALITY CONTROL DATA FROM 20/01/82 TO 30/12/82

LAB: Rivers and Lakes

Analytical Range: 0.32 to 100.0 mg/L as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	100	40.0	40.2	0.2	0.49
B :	100	15.0	15.1	0.1	0.30
A+B :	99	55.0	55.2	0.2	0.64
A-B :	99	25.0	25.1	0.1	0.49
C :	105	15.00	15.09	0.09	0.211
D :	106	5.00	5.07	0.07	0.124
C+D :	104	20.00	20.15	0.15	0.269
C-D :	104	10.00	10.01	0.01	0.214

s.d(AB): Sw(within run): 0.35 S(between runs): 0.41 S/Sw: 1.16
s.d(CD): Sw(within run): 0.151 S(between runs): 0.173 S/Sw: 1.15

On any given day the calibration is accepted if the values obtained lie within the ranges:

50.5 to 59.5 for A+B
22.0 to 28.0 for A-B
19.10 to 20.90 for C+D
9.40 to 10.60 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
6	0.00 - 2.00	0.195	19.46
171	2.00 - 10.00	0.210	3.50
26	10.00 - 20.00	0.549	3.66
24	20.00 - 50.0	1.17	3.33
1	50.0 - 100.0	N/A	N/A
228	Overall	0.54	N/A

DETECTION CRITERION: 0.32

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	106	-0.00	0.226

SULPHATE

QUALITY CONTROL DATA FROM 11/01/83 TO 19/12/83

LAB: Rivers and Lakes

Analytical Range: 0.13 to 100.0 mg/L as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	135	40.0	40.5	0.5	0.49
B :	136	15.0	15.0	0.0	0.32
A+B :	134	55.0	55.5	0.5	0.72
A-B :	134	25.0	25.4	0.4	0.39
C :	136	15.00	15.14	0.14	0.202
D :	135	5.00	5.04	0.04	0.115
C+D :	134	20.00	20.18	0.18	0.255
C-D :	134	10.00	10.09	0.09	0.199

s.d(AB): Sw(within run): 0.28

S(between runs): 0.41

S/Sw: 1.50

s.d(CD): Sw(within run): 0.141

S(between runs): 0.164

S/Sw: 1.17

On any given day the calibration is accepted if the values obtained lie within the ranges:

50.5 to 59.5 for A+B

22.0 to 28.0 for A-B

19.10 to 20.90 for C+D

9.40 to 10.60 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
23	0.00 - 2.00	0.080	7.97
253	2.00 - 10.00	0.196	3.26
56	10.00 - 20.00	0.404	2.69
38	20.00 - 50.0	1.58	4.51
0	50.0 - 100.0	N/A	N/A
370	Overall	0.57	N/A

DETECTION CRITERION: 0.13

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	135	0.00	0.000

SULPHATE

QUALITY CONTROL DATA FROM 04/01/84 TO 04/12/84

LAB: Rivers and Lakes

Analytical Range: 0.23 to 100.0 mg/L as SO₄

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	176	60.0	60.0	0.0	0.71
B :	176	15.0	15.1	0.1	0.27
A+B :	175	75.0	75.1	0.1	0.80
A-B :	175	45.0	45.0	-0.0	0.72
C :	176	15.00	15.06	0.06	0.188
D :	176	5.00	4.99	-0.01	0.084
C+D :	175	20.00	20.05	0.05	0.221
C-D :	175	10.00	10.08	0.08	0.191

s.d(AB): Sw(within run): 0.51 S(between runs): 0.54 S/Sw: 1.06
s.d(CD): Sw(within run): 0.135 S(between runs): 0.146 S/Sw: 1.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

70.5 to 79.5 for A+B
42.0 to 48.0 for A-B
19.10 to 20.90 for C+D
9.40 to 10.60 for C-D

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
15	0.00 - 2.00	0.138	13.82
205	2.00 - 10.00	0.261	4.34
52	10.00 - 20.00	0.497	3.31
32	20.00 - 50.0	0.84	2.39
8	50.0 - 100.0	1.93	2.57
312	Overall	0.54	N/A

DETECTION CRITERION: 0.23

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	161	0.01	0.004

*** SULPHUR DIOXIDE ***

IDENTIFICATION:

Laboratory : Precipitation Method Introduced: 01/07/80
LIS Test Name Code: SS02FR Units : ug/Filter as SO2
Work Station Code : PRSEQ,PRLOV Unit Code : 361943
Method Code : 004AIO Supervisor : M. Rawlings
Sample Type/Matrix: Impregnated W41 filters from sequential and LoVol filter packs.

SAMPLING:

Quantity Required: 1 filter
Container : Polyethylene bags
Other : Filter is impregnated with potassium carbonate/glycerol solution.

SAMPLE PREPARATION:

Filters are extracted with 50 ml. of 0.05% H2O2 in polystyrene tubes with 1 hour of mechanical shaking, followed by ultrasonic treatment to enhance extraction, then a 24 hour rest period. SO2 is converted to SO4 in the process.

ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO4 is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as SO2.
Full scale conductivity : 10 uS/cm.

INSTRUMENTATION:

-Mechanical shaker; ultrasonic bath; polyethylene tubes
-Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction and timing.

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 1.65 ug/filter Detection Criterion (T): 6 ug/filter

CALIBRATION:

BL plus 6 standards

CONTROLS:

Calibration : 2 standards, eg, QCA
Drift : 1 standard every 10 samples

MODIFICATIONS:

01/07/80 - Ion chromatographic procedure for precipitation samples was modified for analysis of W41 filter extracts by developing the extraction procedure.
10/03/84 - Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced.
15/03/84 - Streamlined procedure for extraction of W41 filters in one 50 mL polyethylene tube was adopted, eliminating two container transfers, and changing the extraction volume to 50.0 mL from 100.0 mL.

NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

SULPHUR DIOXIDE

QUALITY CONTROL DATA FROM 04/01/82 TO 01/12/82

LAB: Precipitation

Analytical Range: 9.0 to 700 ug/filter as SO₂

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	65	533.3	532.9	-0.4	11.87
B :	65	133.3	134.4	1.0	4.93
A+B :	64	666.7	668.0	1.4	11.28
A-B :	64	400.0	399.4	-0.6	11.34

s.d(AB): Sw(within run): 8.02 S(between runs): 9.09 S/Sw: 1.13

On any given day the calibration is accepted if the values obtained lie within the ranges:

636.7 to 696.7 for A+B
380.0 to 420.0 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
17	0.0 - 200.0	5.47	5.47
4	200.0 - 300.0	7.20	2.88
7	300.0 - 700	7.3	1.46
28	Overall	6.5	N/A

DETECTION CRITERION: 9.0

SULPHUR DIOXIDE

QUALITY CONTROL DATA FROM 10/01/83 TO 06/03/84

LAB: Precipitation

Analytical Range: 11.8 to 700 ug/filter as SO₂

CALIBRATION CONTROL:

	Number of Data	Expected Concn	Av. Concn Measured	Av. Bias	Standard Deviation
A :	62	533.3	536.2	2.9	7.50
B :	89	133.3	144.4	11.1	8.39
A+B :	62	666.7	681.2	14.5	11.63
A-B :	62	400.0	391.3	-8.7	12.05

s.d(AB): Sw(within run): 8.52

S(between runs): 7.96

S/Sw: 0.93

On any given day the calibration is accepted if the values obtained lie within the ranges:

636.7 to 696.7 for A+B
380.0 to 420.0 for A-B

DUPLICATES:

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
33	0.0 - 200.0	7.21	7.21
4	200.0 - 300.0	16.33	6.53
0	300.0 - 700	N/A	N/A
37	Overall	8.9	N/A

DETECTION CRITERION: 11.8

SULPHUR DIOXIDE

QUALITY CONTROL DATA FROM 11/05/84 TO 27/12/84

LAB: Precipitation

Analytical Range: 5.5 to 350 ug/filter as SO₂

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	91	266.7	267.4	0.8	3.16
B :	91	66.7	67.3	0.7	1.74
A+B :	91	333.3	334.7	1.4	3.72
A-B :	91	200.0	200.1	0.1	3.48

s.d(AB): Sw(within run): 2.46

S(between runs): 2.55

S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

318.3 to 348.3 for A+B
190.0 to 210.0 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
96	0.0 - 100.0	3.33	6.67
11	100.0 - 150.0	4.04	3.23
20	150.0 - 350	8.3	3.32
127	Overall	4.9	N/A

DETECTION CRITERION: 5.5

*** TURBIDITY ***

IDENTIFICATION:

Laboratory	: Domestic Water	Method	: Before '74
LIS Test Name Code	: TURB	Units	: FTU
Work Station Code	: WTURB	Unit Code	: 343000
Method Code	: 002A11	Supervisor	: M. Rawlings
Sample Type/Matrix	: Domestic Water, Leachates, Effluents		

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

The instrument is standardized with a sealed standard which is prepared commercially from latex polymers of known size and rated in Formazin Turbidity Units. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurement are based on light scattering at 90 plus or minus 30 degrees of rotation. The instrument compensates for sample colour.

INSTRUMENTATION:

Hach Ratio 18900 Turbidimeter

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 0.01 Detection Criterion (T): 0.4

CALIBRATION:

BL plus 1 standard.

CONTROLS:

Calibration: None

MODIFICATIONS:

01/03/84-Hach 2100A turbidimeter was replaced by Hach ratio turbidimeter.

NOTES:

In the past samples were not stirred during turbidity measurements in the Domestic Water laboratory even though the former instrument (Hach 2100A) possessed this capability. Thus the effect of changing the instrumentation was minimal.

TURBIDITY

QUALITY CONTROL DATA FROM 04/01/82 TO 18/12/84

LAB: Domestic Water

Analytical Range: 0.5 to 200 FTU

DUPLICATES: (1982)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
377	0.0 - 10.0	0.18	3.68
17	10.0 - 25.0	0.47	2.69
6	25.0 - 50.0	1.03	2.75
2	50.0 - 100	0.0	0.00
0	100 - 200	N/A	N/A
402	Overall	0.6	N/A

DETECTION CRITERION: 0.3

DUPLICATES: (1983)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
318	0.0 - 10.0	0.21	4.23
10	10.0 - 25.0	1.02	5.82
7	25.0 - 50.0	0.69	1.84
1	50.0 - 100	N/A	N/A
1	100 - 200	N/A	N/A
337	Overall	0.5	N/A

DETECTION CRITERION: 0.3

DUPLICATES: (1984)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
255	0.0 - 10.0	0.33	6.65
25	10.0 - 25.0	0.53	3.03
10	25.0 - 50.0	1.95	5.19
6	50.0 - 100	0.9	1.19
5	100 - 200	1.8	1.19
301	Overall	0.6	N/A

DETECTION CRITERION: 0.5

*** TURBIDITY ***

IDENTIFICATION:

Laboratory	: Rivers and Lakes	Method Introduced:	01/04/74
LIS Test Name Code:	TURB	Units	: FTU
Work Station Code	: RMTURB	Unit Code	: 343000
Method Code	: 002A11	Supervisor	: J. Crowther
Sample Type/Matrix:	Rivers, Lakes, Effluents		

SAMPLING:

Quantity Required: 50 mL
Container : Glass or plastic

ANALYTICAL PROCEDURE:

The instrument is standardized with a sealed standard which is prepared commercially from latex polymers of known size and rated in Formazin Turbidity Units. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurement are based on light scattering at 90 plus or minus 30 degrees of rotation. The instrument compensates for sample colour.

INSTRUMENTATION:

- Hach Ratio 18900 Turbidimeter

REPORTING:

Maximum Significant Figures: 3	
Minimum Increment (W) : 0.01	Detection Criterion (T): 0.4

CALIBRATION:

BL plus 1 standard.

CONTROLS:

Calibration: None

MODIFICATIONS:

01/04/82-Hach 2100A turbidimeter was replaced by Hach ratio turbidimeter. As of this date samples are no longer stirred during turbidity measurements, and thus the effect of heavy particulates is minimized as they settle out before the reading is accepted.

TURBIDITY

QUALITY CONTROL DATA FROM 04/01/82 TO 31/12/84

LAB: Rivers and Lakes

Analytical Range: 0.4 to 100.0 FTU

DUPLICATES: (1982)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
295	0.00 - 5.00	0.071	2.85
105	5.00 - 10.00	0.082	1.10
93	10.00 - 25.0	0.21	1.19
55	25.0 - 50.0	0.50	1.33
24	50.0 - 100.0	0.62	0.83
572	Overall	0.24	N/A

DETECTION CRITERION: 0.12

DUPLICATES: (1983)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
287	0.00 - 5.00	0.196	7.85
80	5.00 - 10.00	0.226	3.02
99	10.00 - 25.0	0.81	4.61
30	25.0 - 50.0	1.02	2.72
26	50.0 - 100.0	1.27	1.69
522	Overall	0.67	N/A

DETECTION CRITERION: 0.32

DUPLICATES: (1984)

Number of Data Pairs	Sample Concn Span	Mean s.d.	Relative s.d. (%)
289	0.00 - 5.00	0.231	9.23
102	5.00 - 10.00	0.375	5.00
98	10.00 - 25.0	0.88	5.00
32	25.0 - 50.0	4.82	12.86
27	50.0 - 100.0	0.99	1.31
548	Overall	1.35	N/A

DETECTION CRITERION: 0.38

*** VOLATILE ACIDS ***

IDENTIFICATION:

Laboratory	: Sewage/Industrial	Method Introduced:	Before '74
LIS Test Name Code:	VOLACF	Units	: mg/L as HOAC
Work Station Code	: VA	Unit Code	: 064705
Method Code	: 101AT0	Supervisor	: P. Campbell
Sample Type/Matrix:	Digested Sludge (anaerobic)		

SAMPLING:

Quantity Required: 25 mL
Container : Glass

ANALYTICAL PROCEDURE:

Sludge are filtered and an acidified aliquot of the supernatant is pipetted on a column of granular silicic acid. A chloroform/n-butanol solvent is passed through the column, and low molecular weight organic acids are preferentially eluted. The eluent is collected and titrated with a standard solution of alcoholic 0.02 N sodium hydroxide to the phenolphthalein endpoint.

INSTRUMENTATION:

Short glass chromatographic columns, manual titration assembly

REPORTING:

Maximum Significant Figures: 3
Minimum Increment (W) : 2 Detection Criterion (T): 30

CALIBRATION:

BL plus normality of sodium hydroxide is verified by titrating with potassium hydrogen phthalate.

CONTROLS:

Calibration : 2 standards, eg, QCA

MODIFICATIONS:

25/06/84- Digital burette (readability to 0.01 mL) replaced glass burette.

VOLATILE ACIDS

QUALITY CONTROL DATA FROM 11/01/82 TO 14/12/82

LAB: Sewage/Industrial

Analytical Range: 8 to 2000 mg/L as Acetic

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	49	1000	1025	25	22.0
B :	49	100	106	6	8.6
A+B :	49	1100	1132	32	25.2
A-B :	49	900	919	19	22.0

s.d(AB): Sw(within run): 15.5

S(between runs): 16.7

S/Sw: 1.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

988 to 1213 for A+B
825 to 975 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
9	0 - 200	5.2	5.18
7	200 - 400	6.0	2.00
28	400 - 1000	6.6	0.95
1	1000 - 2000	N/A	N/A
45	Overall	6.2	N/A

DETECTION CRITERION: 8

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	49	142	16.4

VOLATILE ACIDS

QUALITY CONTROL DATA FROM 12/01/83 TO 29/12/83

LAB: Sewage/Industrial

Analytical Range: 22 to 2000 mg/L as Acetic

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	46	1000	1046	46	42.0
B :	46	100	108	8	16.1
A+B :	46	1100	1154	54	50.9
A-B :	46	900	939	39	38.2

s.d(AB): Sw(within run): 27.0

S(between runs): 31.8

S/Sw: 1.18

On any given day the calibration is accepted if the values obtained lie within the ranges:

988 to 1213 for A+B
825 to 975 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
17	0 - 200	13.6	13.61
15	200 - 400	31.4	10.46
13	400 - 1000	26.9	3.84
0	1000 - 2000	N/A	N/A
45	Overall	24.2	N/A

DETECTION CRITERION: 22

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	43	137	27.9

VOLATILE ACIDS

QUALITY CONTROL DATA FROM 16/01/84 TO 17/12/84

LAB: Sewage/Industrial

Analytical Range: 29 to 2000 mg/L as Acetic

CALIBRATION CONTROL:

	<u>Number of Data</u>	<u>Expected Concn</u>	<u>Av. Concn Measured</u>	<u>Av. Bias</u>	<u>Standard Deviation</u>
A :	43	1000	1041	41	35.2
B :	42	100	105	5	10.2
A+B :	42	1100	1147	47	37.2
A-B :	42	900	936	36	37.0

s.d(AB): Sw(within run): 26.2

S(between runs): 26.0

S/Sw: 0.99

On any given day the calibration is accepted if the values obtained lie within the ranges:

988 to 1213 for A+B
825 to 975 for A-B

DUPLICATES:

<u>Number of Data Pairs</u>	<u>Sample Concn Span</u>	<u>Mean s.d.</u>	<u>Relative s.d. (%)</u>
26	0 - 200	17.9	17.87
8	200 - 400	21.1	7.03
3	400 - 1000	88.9	12.70
1	1000 - 2000	N/A	N/A
38	Overall	31.4	N/A

DETECTION CRITERION: 29

OTHER CHECKS:

	<u>Number of Data</u>	<u>Data Mean</u>	<u>Standard Deviation</u>
Long Term Blank :	43	143	23.7